# EFFECT OF WATER ON EPOXY RESIN'S CURING CHARACTERISTIC

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by

Teh Hui Nee

Dissertation submitted in partial fulfilment of
the requirements for the
Bachelor of Engineering (Hons)
(Chemical Engineering)

July 2009

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CERTIFICATION OF APPROVAL

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Approved by,

(Dr Zakaria Man)

UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

July 2009

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

TEH HUI NEE

# ACKNOWLEDGEMENT

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Last but not least, special thanks to my family and coursemates for their support and encouragement in helping us completing this project for this semester.

Once again, thank you to all of you.

# ABSTRACT

Polymerization will take place in the reaction between resin and hardener. Different conditions and temperature will affect the polymerization reaction. Therefore few methods can be applied to investigate the curing process of resin especially underwater. For example, in sub sea environment, resin is used to repair the defect areas of pipes which are used to transfer gas and petroleum products. Therefore it is very crucial to make sure that the resins are fully cured to resume full capacity operations. Conventional method used is Differential Scanning Calorimeter where the percentage of cure can be calculated but the chemical reaction is not known. Infrared and Raman analysis are the appropriate methods to observe the chemical reaction of resin. Since Raman spectroscopy is currently under maintenance, in order to investigate the effect of water to curing process by using infrared analysis. There are many functional groups can be detected by the infrared, in this experiment, epoxide and phenylene groups are essential for the observation. The epoxy resin used is EPOLAM 2050 which is used in concrete and in casting by addition of fillers. The experimental method is carried out by considering the properties of this resin which is quite viscous. There are plenty ways to determine the effect of water on epoxy resin. However, only two ways are implemented here which are adding the water with resin and another is immerse the resin plate into water. For effect of temperature, conventional way is applied where the samples are cured in a oven. To analyse the results chemically and mechanically, quite a number of equipment can be applied but due to time consumption and availability of equipments, only Fourier Transform Infrared Spectroscopy is used. From this analysis, it can clearly show the effect of water and temperature on resin. When both parameters increase, there curing rate will increase too. So the relation of water and temperature without exceeding the limit is directly proportional to the curing. Overall, the objectives of the research are achieved but there are room for improvement and recommendation to expand it.

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# CHAPTER 1
# INTRODUCTION

## 1.1 Background of Study

Epoxy resin is a high performance thermosetting resin. It is a polymeric material and has been commercially available for almost a half century. The outstanding physical properties exhibited by epoxy resin include:

a) Low cure shrinkage
b) No volatile given off during curing process
c) Compatibility with many materials
d) Adhesion
e) Corrosion and chemical resistance
f) Electrical insulation

Epoxy resin is useful and widely applied in various applications such as repairing and coatings in composite matrices, filling wide cracks, gaps and delamination in concrete. It is commercially available in liquid and solid form. The liquid is available as solvent free resin, waterborne emulsions and solvent borne solutions. Basically, epoxy resin composed of polymeric molecules which are converted to solid by a chemical reaction. Epoxy system physically consists of two main components which are resin and curing agent. The curing agent or known as hardener causes the chemical reaction to occur, turning the epoxy resin into a solid and crosslinked network of molecules. This type of polymer is categorized as thermoset polymer because when it cured, it is an irreversible rigid and relatively unaffected by heat.

The main concern in epoxy resin is the curing process or also known as polymerization. This epoxy system is capable to cure at either ambient or elevated temperature with minimal pressure requirement. In ambient temperature, the curing duration is predictable based of Material Safety Data Sheet. If the resin is cured at different conditions, the curing duration need to be observed. The condition that the author studied is when resin at underwater condition or in humid environment. Nowadays, epoxy resin is also widely used in repairing pipeline at subsea level. Therefore, it is essential to know the curing process and duration taking place in watery condition compared to the ambient temperature before the operation in subsea level can be resumed.

## 1.2 Problem Statement

The components to be identified in the project initially are the problem identification and the significant of the project.

### 1.2.1 Problem Identification

For technical structure with epoxy resin, the resin must be fully cured and very stable attached on it. This is essential in repairing work in order to avoid leakage and to ensure safe operation. Different type of resin will have own specified curing time and properties. At various conditions such as temperature, curing environment and ratio of chemical, the polymerization process will not obey to the normal process occurs during standard conditions. The curing rate will be affected. Therefore, during this situation, information available in Material Safety Data Sheet could not be referred. The properties of resin must be well known before applying on technical structure. However, the related information regarding the curing characteristic and chemical reaction of epoxy resin humid environment is limited. Thus, to obtain complete information of epoxy resin in term of chemical and mechanical, combination analysis on chemical and mechanical must be carried out on epoxy resin.

### 1.2.2. Significance of the Project

When people apply epoxy resin on concrete or any other structure, it is very important to ensure that the resin is fully cured before applying it on other structure. Nowadays, in order to save the cost and time, pipelines which are used for transferring oil and gas are repaired by using this method, which is by applying epoxy resin. In the pipeline, the temperature of oil and gas might be very high and affect the curing of resin. Other than that, for pipeline at subsea, the curing of resin will be affected. Therefore, with the curing duration known, workers can predict when to fully resume the pipeline's operation.

## 1.3 Objectives and Scope of Study

### 1.3.1 Objectives

The purposes of this research project are:

a) To monitor the curing of epoxy resins by using an effective spectroscopy analysis.

b) To study the water absorption on chemical reaction of resins underwater.

c) To compare the curing characteristic for resins at different temperature.

The initial planning of this project is to apply Raman analysis to study on the water effect on epoxy resin. However, due to some maintenance work on Raman spectroscopy, infrared is used to replace Raman analysis. In convention method, infrared can be easily absorbed by water and make it not relevant in analyzing wet resin samples. Therefore, sample preparation is an important step in order to determine the water effect on resin.

### 1.3.2 Scope of Study

The main subject to be studies in this research is epoxy resin. As the main tool to analyse the curing characteristic, Fourier Transform Infrared Spectroscopy (FTIR) for both solid and liquid sample is used. Initially the infrared analysis is to be used together with Raman analysis to get more convincing results. There is no research on epoxy resin at underwater condition analysed by Raman in the market.

# CHAPTER 2

# LITERATURE REVIEW AND/OR THEORY

## 2.1 Overview

Generally, the epoxy resin widely in use is Diglycidyl Ether of Bisphenol A ((4-(2,3 epoxypropoxy) phenyl) propane) which is also known as DGEBA [1]. Meanwhile, the hardeners are aliphatic or aromatic amines which play the role as curing agent. The epoxy resins and amines might be in different chemical structures, but the chemical reaction that involves is similar. The main structures that involve in polymerization are the epoxide and amine group. Figure 1 shows the reaction of epoxy resin with amine in polymerization process. Figure 2 is the chemical structure of epoxide.

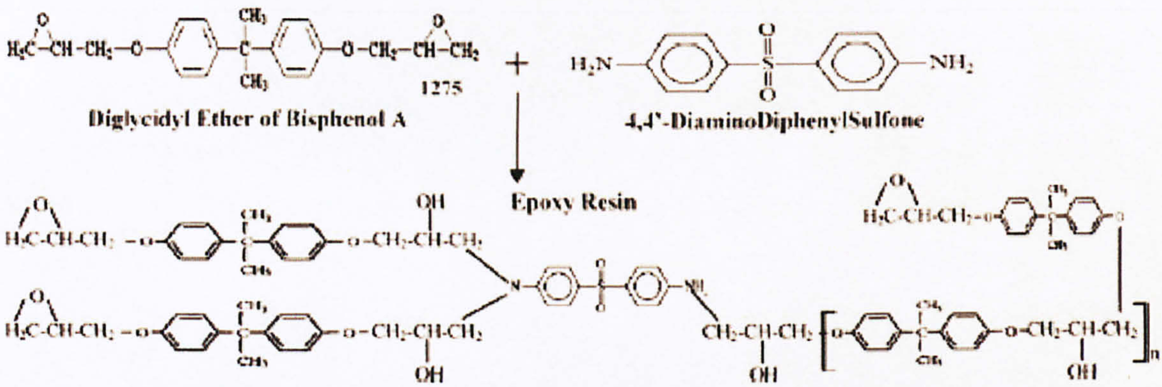



**Figure 1:** Polymerization process of epoxy resin with hardener

Epoxide is a group with three membered ring consists of oxygen and the two carbons. It is also called an oxirane ring, or the glycidyl group [2].

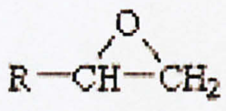


**Figure 2:** Chemical structure of epoxide

In this project the epoxy resin used is EPOLAM 2050. This type of resin also consists of resin and hardener. The resin is made up of bisphenol A-(epichlorhydrin) in Figure 3 and tetraglycidylether methylene bis aniline in Figure 3.

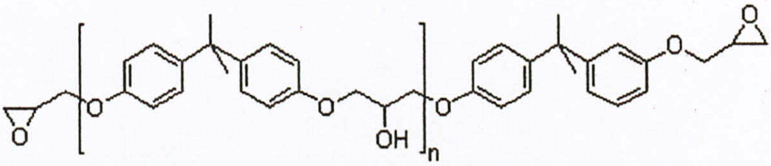


**Figure 3:** tetraglycidylether methylene bis aniline

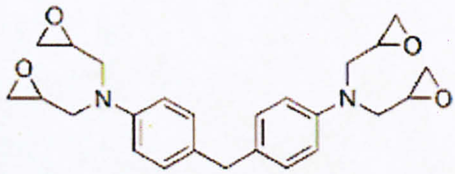


**Figure 4:** tetraglycidylether methylene bis aniline

For hardener, the compositions are *3*-aminomethyl-3,5,5-trimethylcyclohexylamine, polyoxyalkyleneamine and diethylmethylbenzenediamine. These hardeners are formed by aromatic, aliphatic and cycloaliphatic amines as indicated in figures below. With combination of various amine, the hardener will have different properties with other type of amine.

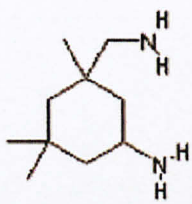


**Figure 5:** *3*-aminomethyl-3,5,5-trimethylcyclohexylamine

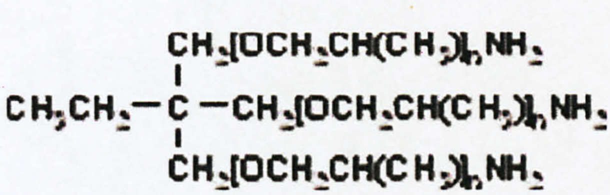


**Figure 6:** polyoxyalkyleneamine

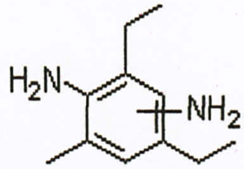


**Figure 7:** diethylmethylbenzenediamine

Polymerization process or also known as crosslinking is a chemical process where the epoxy and amine will react together to form a material with identified strength and adhesion. As the curing goes on, epoxide group will be reduced as it is the main group that has chemical reaction during the polymerization. The physical and chemical properties of cured resin are determined by:

a) The structure of the resin: number, location and reactivity oepoxy and o ther groups present.

b) The curing agent, stoichiometry and cure conditions.

These factors determined the rigidity, thermal stability, chemical resistance and function of epoxy resin.

## 2.2 Temperature Effect on Epoxy Resin

In principal, the room temperature curing will not be like curing at elevated temperature. At elevated temperature, the epoxy resin and hardener are mobile and have greater potential for reaction than in room temperature. Other than that, the glass transition temperature will be affected too. In some situation, high temperature will degrade the epoxy resin. The table shows the advantages and disadvantages of elevated temperature curing epoxy.

**Table 1:** Advantages and disadvantages of elevated temperature curing epoxy

| Advantages | Disadvantages |
|---|---|
| • Fast cure time results in higher production speed and less tie-up of fixturing equipment<br>• Long working life<br>• High temperature and chemical resistance<br>• Long shelf life for two system components<br>• Solid(film, powder) systems possible<br>• Viscosity decreases at elevated temperature provide more efficient wetting of substract | • Greater internal stresses than in room temperature curing because:<br>- Higher shrinkage on polymerization<br>- Thermal expansion coefficient differences<br>• More brittle (poorer peel and impact properties)<br>• Safety and hazardous nature of high temperature<br>• Energy consumption<br>• Viscosity decreases could result in starved joint |

## 2.3 Water Effect on Epoxy Resin

Water is a very polar and permeates easily into most polymers, including epoxy resin. Epoxy adhesive is not immune to water attack eventhough insoluble in water. Hydrolysis process will break the bonds of epoxy and amine. If the physical properties can be tested, moisture attack will cause swelling and increasing of internal stress which will lead to debonding. It also can affect the glass transition temperature of epoxy resin when it chemically reacts with the polymer. Water permeation lowers the glass transition temperature of epoxy resin by reducing the attractive forces between molecules. [3] Table 1 shows the effect of water on mechanical properties of epoxy resin.

**Table 2:** Effect of water on mechanical properties of epoxy resin

| **Exposure conditions** | **Weight gain,%** | **Tensile strength, MPa** | **Elongation at break, %** | **Modulus, MPa** | **Failure mode** |
|---|---|---|---|---|---|
| Epoxy/polyamide | | | | | |
| None | 0 | 73 | 5 | 1880 | Brittle |
| 3months at 65% RH | 2.9 | 52 | 263 | 623 | Ductile |
| 5 days in water at 50°C | 9.4 | 19 | 260 | 3 | Rubbery |
| 5 days in water at 50°C, then dried at 60°C for 2 days | 3.3 | 76 | 5.7 | 1980 | Brittle |

In some situation, the absorbed water on shows no chemical linkages are broken, it means the properties is fully recover when the polymer dried. Amine cured epoxy will have better hydrolytic stability compared to others such as anhydride cured. Figure 3 illustrates the degradation of polymer chains by hydrolytic reaction with water.

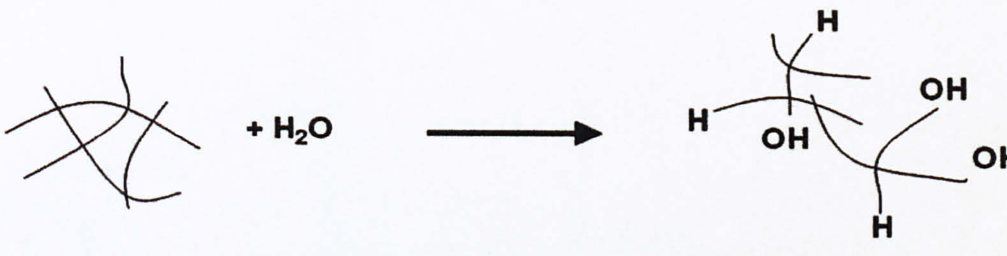



**Figure 8:** Degradation of polymer chain by hydrolysis process

For uncured epoxy system, the reactivity of the resin is dependant on the number of reactive epoxy groups on the molecules. Other than epoxy rings, hydroxyl group might presence in the molecules. Hydroxyl groups can be contributed by the water. It can affect the reactivity and polymerization process. The number and location could either accelerate or retard the overall reaction rate and lead to different three dimensional cured polymeric structure.

For crosslinking process that occurs at cool ambient temperature or high humidity, this will develop a surface oiliness, exudate or whitish spots variously referred to as "amine blush". Blushing is caused by absorption of moisture and carbon dioxide from the atmosphere during curing process. The amine compounds used for epoxy curing are often both hygroscopic and very efficient scavengers of carbon dioxide from the air, eventhough carbon dioxide presents at levels averaging only about 350 ppm in outdoor atmospheres. In indoor environments with human activity, the amount of carbon dioxide present may increase by two or three times. Although overlooked by many end-users of epoxy resins, amine compounds used as epoxy curing agents readily scavenge carbon dioxide (and sometimes moisture) from the atmosphere. Therefore, minimizing the air exposure time of uncured amine/epoxy resin formulations can decrease the appearance of problems related to blush formation. [4]

Rinker, Ashour, and Sandall proposed and provide many references for, the reaction schemes described in Figure 7 below. Primary and secondary amines will rapidly react with carbon dioxide to form carbamate zwitterions. With the addition of water, the absorption capacity will increase. One mole of carbon dioxide will react with two moles of amine. Tertiary amines could not react directly with carbon dioxide, but in aqueous solutions they promote the hydrolysis of carbon dioxide, forming protonated amine bicarbonates. The stoichiometry is 1:1, allowing greater sorption of carbon dioxide. Illustrations of such reactions are as Figure 4.

$$R_1R_2NH + CO_2 \rightleftharpoons R_1R_2NH^+COO^-$$

Primary or secondary amines — Carbamic acid zwitterion

$$R_1R_2NH^+COO^- + R_1R_2NH \rightleftharpoons R_1R_2NCOO^- + R_1R_2NH_2^+$$

zwitterion — an ammonium carbamate

$$R_1R_2R_3N + CO_2 + H_2O \rightleftharpoons R_1R_2R_3NH^+ + HCO_3^-$$

tertiary amine — an ammonium bicarbonate

**Figure 9:** Reaction of amines with carbon dioxide

There are few ways to eliminate this blushing problem such as decreases the gel time. By shorten the gel time of amine epoxy formulations, the concentration of primary and secondary amines present at the surface that react with carbon dioxide can be limited. The resins can also cured at higher temperature to eliminate the reaction of amine and carbon dioxide. Therefore, process conditions such as storage, mixing, induction time, application method and ambient environmental conditions are important for blush prevention than since they can greatly affect carbon dioxide and moisture sorption of the curing formulation. However, in actual condition, this effect is hardly seen.

## 2.4 Raman Spectroscopy

Raman Spectroscopy is a powerful analytical and research tool to study on chemical reaction of resins. It is a light scattering technique where a light's photon interacts with a sample to produce scattered radiation at different wavelength. It can determine molecular motions, especially vibrations by using Raman. Raman band will arise when there is change of polarizability in molecules. From this analysis, much information regarding resin can be determined such as chemical and molecular structures, effect of environment on chemical bonding and etc. This technique is more suitable to be applied on wet resins because the presence of water will not be affecting the results.

In Raman spectroscopy, the vibration of epoxide can be observed at the wavenumber 1275cm[1]. As curing progresses, the amount of epoxide and the peak will decrease indicates the polymerization process take place between resins and hardeners. Therefore the percentage of cure for resins can be calculated by comparing the differences in intensities using formula below. Figure 3 shows the results of polymer curing at different time interval. [5]

$$\%cure = \frac{I_o - I_t}{I_o}$$

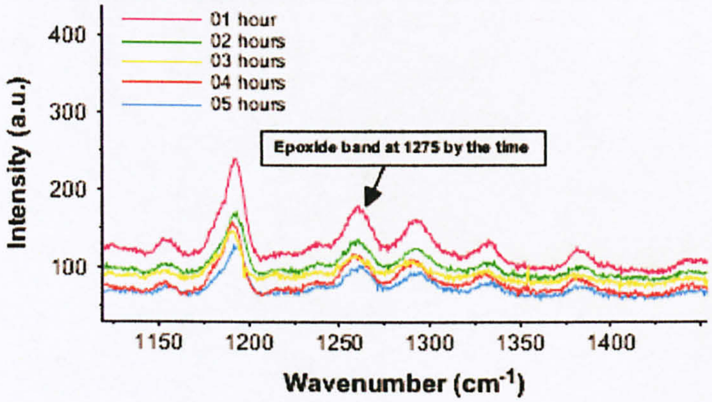



**Figure 10:** Epoxide band at various time

## 2.5 Fourier Transform Infrared Spectroscopy

Infared analysis is a technique to identify unknown samples, quality of samples and amount of components of a mixture from the vibration of the chemical bonds. An IR spectroscopy consists of an energy source, a sample holder, detector and plotter. The background spectrum is obtained and stored before the sample spectrum. The software will then subtract the background spectrum to give the sample spectrum. The sample spectrum is then Fourier transformed to give the final spectrum. The infrared spectrum can be divided into three regions: the far infrared (400-0cm$^{-1}$), the mid infrared (4000-400cm$^{-1}$) and the near infrared (14285-400cm$^{-1}$). Mostly infrared applications apply the mid infrared region, but the near and far infrared region can provide information for certain materials.

The infrared will be absorbed by the sample or transmitted through it. Molecular fingerprint of the sample will be created and analyzed. In this analysis, the composition of epoxy resin can be analyzed. This is to study on the changes in chemical bonding in resin and hardener during crosslinking process. The curing band can be observed at approximately 914cm$^{-1}$ or 5426cm$^{-1}$. By comparing the curing rate, the peaks should be varied for epoxy at different condition. [6]

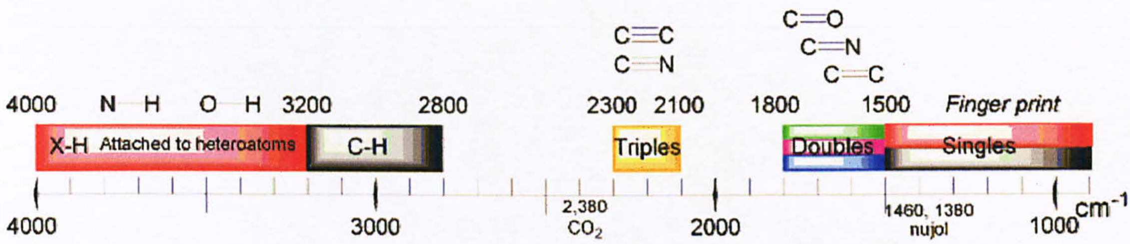



**Figure 11:** Various chemical bond from infrared analysis

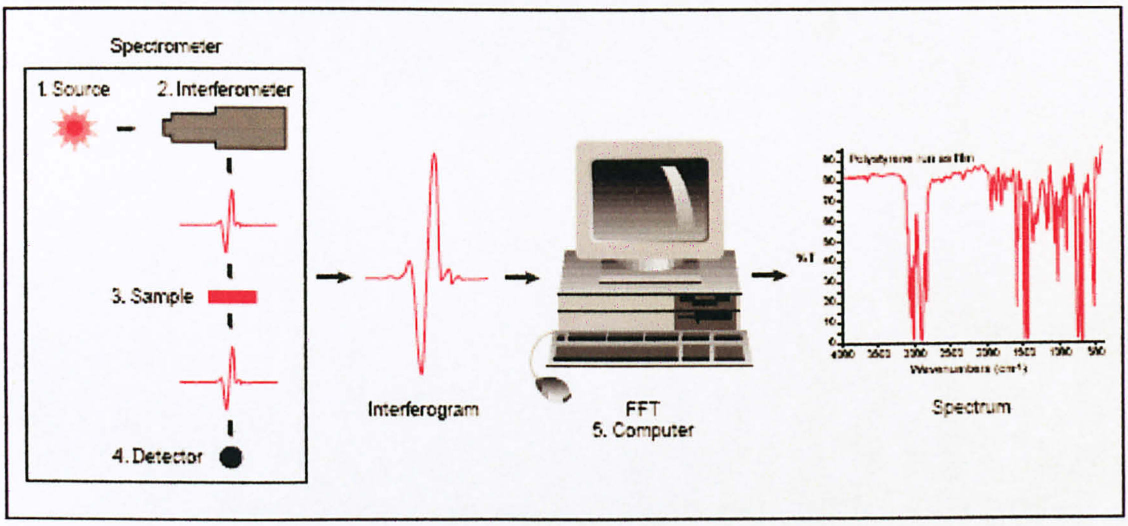



**Figure 12:** Process of infrared analysis works

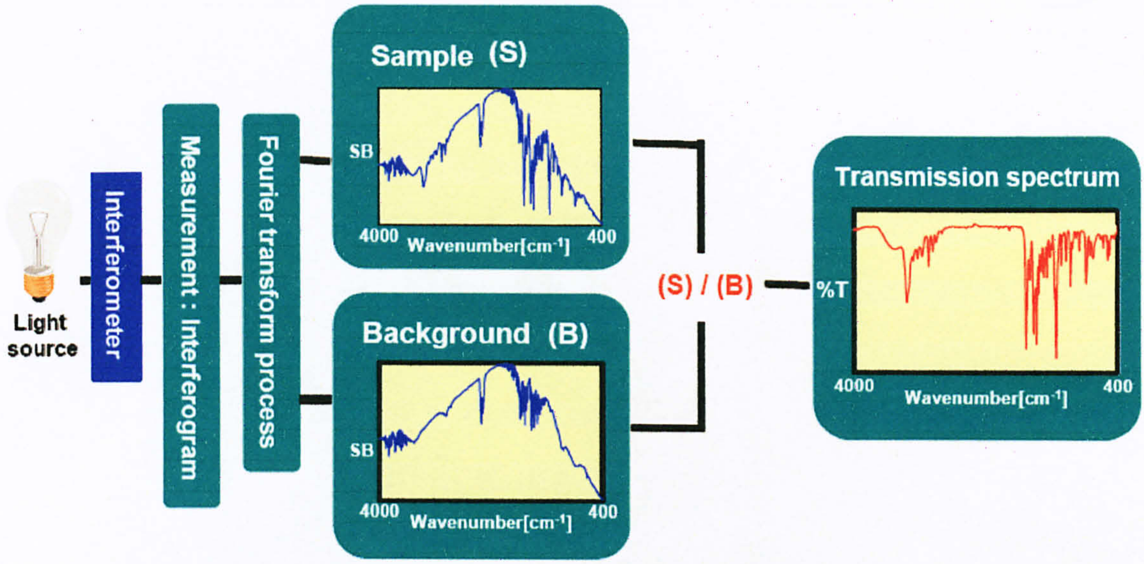



**Figure 13:** Diagram of processing spectrum

There are many methods to prepare the resin sample depends on the nature of sample such as solid or liquid. For solid sample, a simple method is by grinding the sample with KBr powder to form a thin film. Meanwhile, for liquid sample, the liquid resin is directly placed on a KBr cell. However, there are many other ways to prepare it according to the sample properties.

## 2.6 Mechanical Test

A universal testing machine is normally used to test the tensile and compressive properties of material such as polymer. A testing machine consists of a material testing frame, control and analysis software, test fixtures, accessories, parts together with devices to hold and support the sample. Tension test is a destructive test where the sample will finally break or fracture into two pieces. Load is applied on the sample in order to break it. Throughout the tests, control system and its associated software will record the load and extension of the sample. Before breaking, as the area of cross section decreases, large stress is produced. Other than that, the maximum stress that the sample can bear which is called ultimate tensile stress can also be obtained. It can record modulus of elasticity too. With this equipment, complete set of data for mechanical properties of resin can be determined other than chemically.

# CHAPTER 3

# METHODOLOGY

## 3.1 Research Methodology

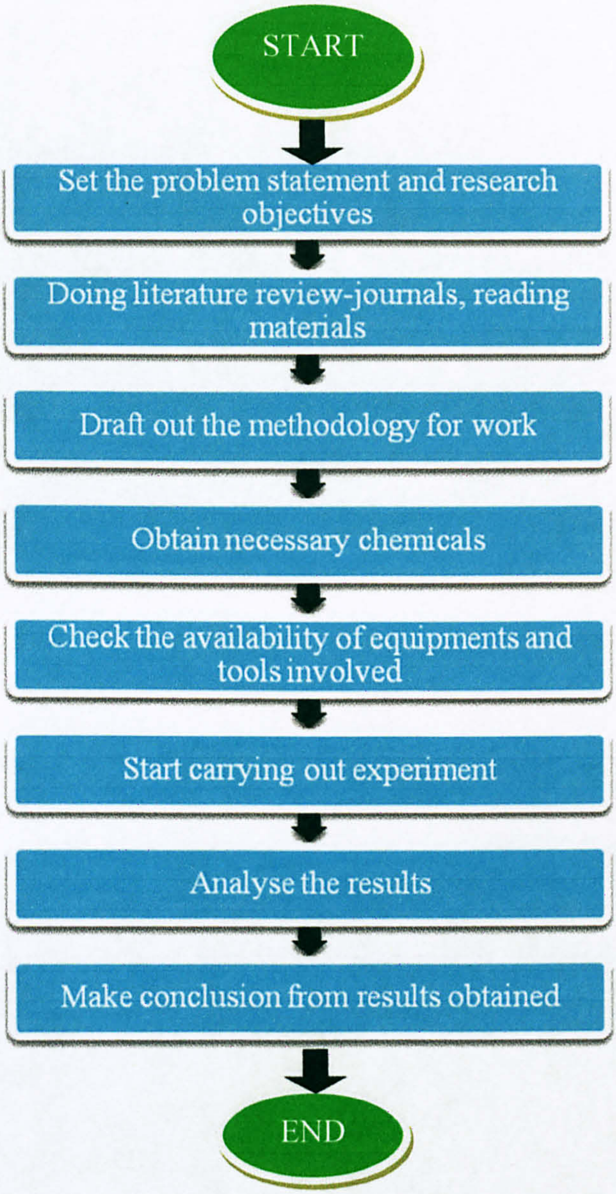



**Figure 14:** Project Flow Chart

## 3.2 Project Schedule

| Project Activities | Week | | | | | | | | | | | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | SEMESTER 1 : January 2009 – June 2009 | | | | | | | | | | | | | | SEMESTER 2 : July 2009 – December 2009 | | | | | | | | | | | | | |
| | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 14 | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 14 |
| Literature Reviews | ■ | ■ | ■ | ■ | ■ | ■ | ■ | ■ | ■ | ■ | ■ | ■ | ■ | ■ | | | | | | | | | | | | | | |
| Literature Analysis | ■ | ■ | ■ | ■ | ■ | ■ | ■ | ■ | ■ | ■ | ■ | ■ | ■ | ■ | | | | | | | | | | | | | | |
| Methodology drafting | | | | | | | | | | | | | | ■ | ■ | ■ | | | | | | | | | | | | |
| Chemical & Equipment Confirmation | | | | | | | | | | | | | | ■ | ■ | ■ | | | | | | | | | | | | |
| Experiment Work | | | | | | | | | | | | | | | | | ■ | ■ | ■ | ■ | ■ | ■ | ■ | ■ | | | | |
| Results Analysis | | | | | | | | | | | | | | | | | ■ | ■ | ■ | ■ | ■ | ■ | ■ | ■ | ■ | | | |
| Discussions and Recommendations | | | | | | | | | | | | | | | | | | | | | | | | ■ | ■ | ■ | | |
| Final Report Documentations | | | | | | | | | | | | | | | | | | | | | | | | | | ■ | ■ | ■ |

| PROJECT MILESTONES | SEMESTER 1 : January 2009 – June 2009 | | | | | | | | | | | | | | SEMESTER 2 : July 2009 – December 2009 | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 14 | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 14 |
| Proposal Submission | | | √ | | | | | | | | | | | | | | | | | | | | | | | | | |
| Preliminary Report | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| Seminar Session | | | | | | | | | √ | | | | | | | | | | | | | | | | | | | |
| Interim Report | | | | | | | | | | | | | | √ | | | | | | | | | | | | | | |
| Oral Presentation | | | | | | | | | | | | | | √ | | | | | | | | | | | | | | |
| Progress Report 1 | | | | | | | √ | | | | | | | | | | | | | | | | | | | | | |
| Progress Report 2 | | | | | | | | | | | | | | | | | | √ | | | | | | | | | | |
| Progress Report 3 | | | | | | | | | | | | | | | | | | | | | | | √ | | | | | |
| Poster Presentation | | | | | | | | | | | | | | | | | | | | | | | | √ | | | | |
| Dissertation (Soft-Bound) | | | | | | | | | | | | | | | | | | | | | | | | | | √ | | |
| Dissertation (Hard-Bound) | | | | | | | | | | | | | | | | | | | | | | | | | | | | √ |
| Final Oral Presentation | | | | | | | | | | | | | | | | | | | | | | | | | | | | √ |

**Figure 15 :** Project Gantt Chart & Key Milestones

## 3.4 Project Activities

Chemical: EPOLAM 2050, CCS Grout

Tool: Fourier Transform Infrared Spectroscopy Shimadzu

### 3.4.1 Effect of Temperature on Epoxy Resin

1. A thin glass is prepared by applying wax on it.
2. Epoxy resin is mixed with hardener at the ratio of 10:3.2.
3. The epoxy mixture is then stirred for 15 minutes with medium speed for well mixing.
4. After mixing, the epoxy is spread uniformly on the glass to form a thin film when it hardens.
5. The glass with epoxy resin is then located in a oven with desired temperature, 50°C.
6. Infrared analysis is tested on resin every 30 minutes.
7. The experiment is repeated for temperature at 120°C and at room temperature which will be analyzed daily.

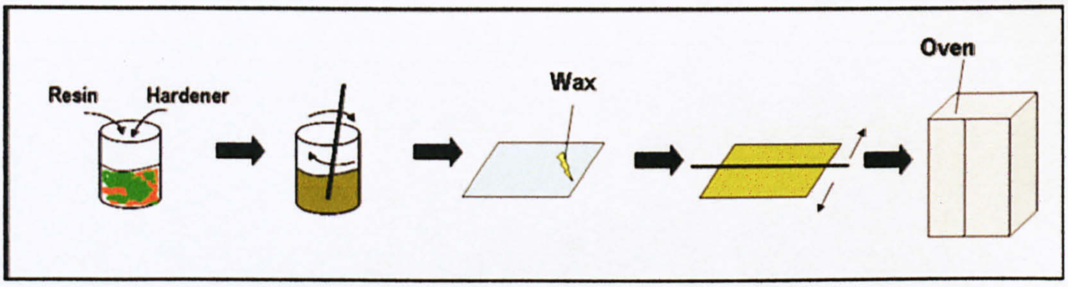



**Figure 16:** Preparation of Sample for Temperature Effect

### 3.4.2 Effect of Water on Epoxy Resin (Part I)

1. A thin glass is prepared by applying wax on it.
2. Epoxy resin is mixed with hardener at the ratio of 10:3.2.
3. The epoxy mixture is then stirred for 15 minutes with medium speed for well mixing.

4. After mixing, the epoxy is mixed with 1% of water and spread uniformly on the glass to form a thin film when it hardens.
5. The glass with epoxy resin is then located at room temperature until it hardens.
6. Infrared analysis is tested on resin the next day after casting.
7. The experiment is repeated for amount of water 2% - 5% at room temperature.

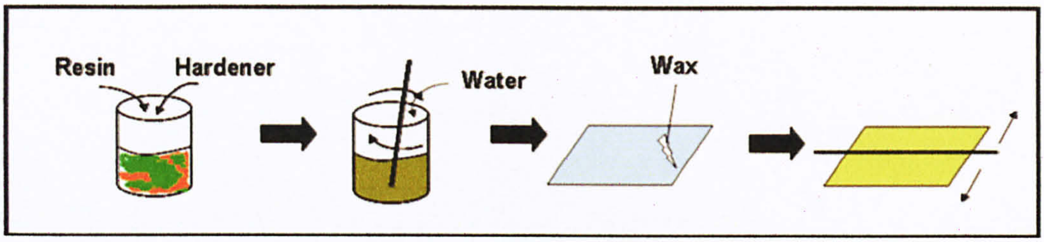



**Figure 17:** Preparation of Sample for Water Effect (I)

### 3.4.3 Effect of Water on Epoxy Resin (Part II)

1. A thin glass is prepared by applying wax on it.
2. Epoxy resin is mixed with hardener at the ratio of 10:3.2.
3. The epoxy mixture is then stirred for 15 minutes with medium speed for well mixing.
4. After mixing, the epoxy is inserted into container which filled with water.
5. The glass with epoxy resin is then located at room temperature until it hardens.
6. Infrared analysis is tested on resin the next day after casting.
7. A dry epoxy is prepared and located in room temperature as control

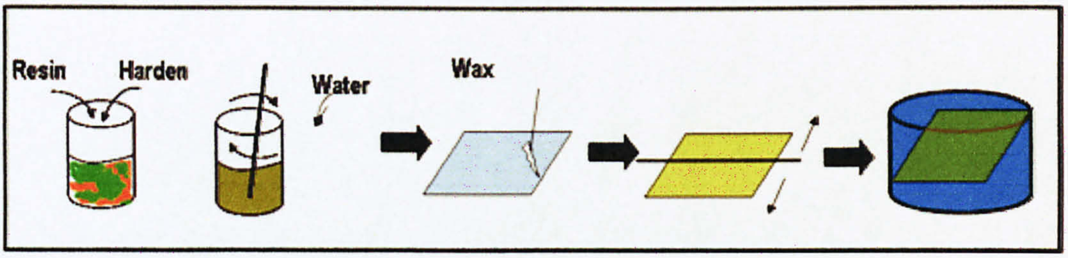



**Figure 18:** Preparation of Sample for Water Effect (II)

# CHAPTER 4

# RESULTS & DISCUSSION

## 4.1 Results

### 4.1.1 Effect of Temperature on Epoxy Resin

#### 4.1.1.1 Temperature at Room

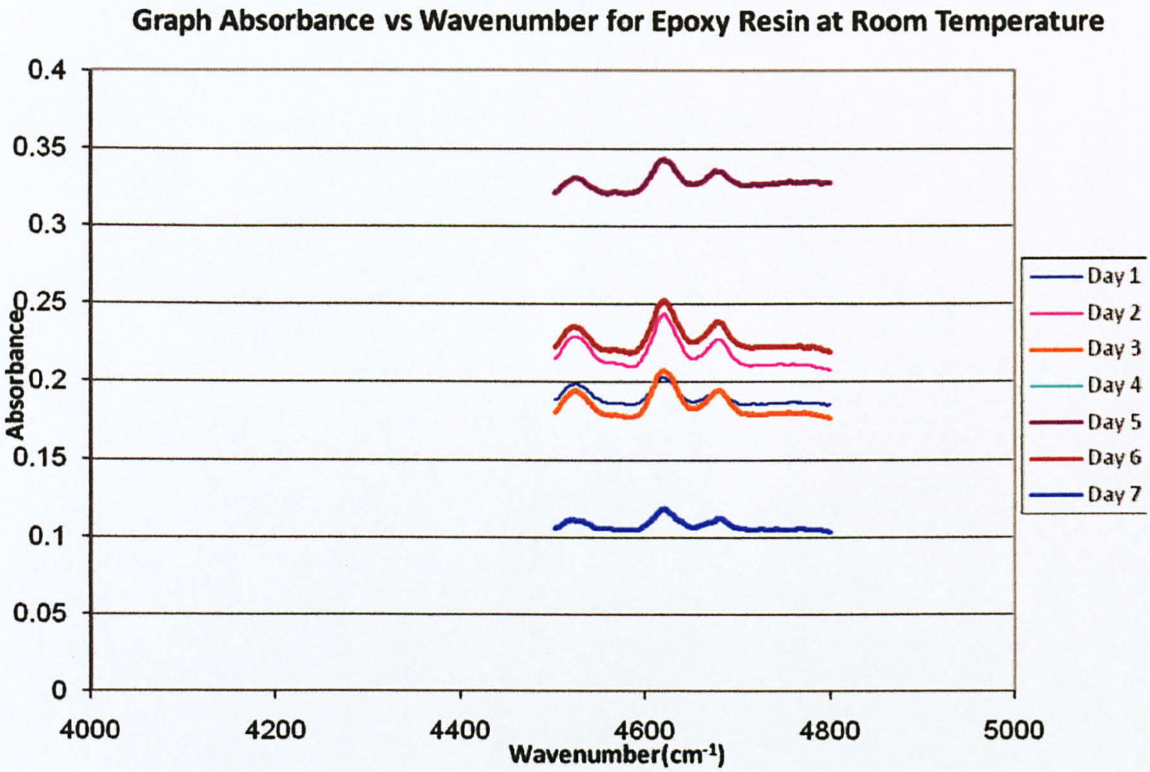



**Figure 19:** Graph for epoxy resin at room temperature at near infrared

### 4.1.1.2 Temperature at 50°C

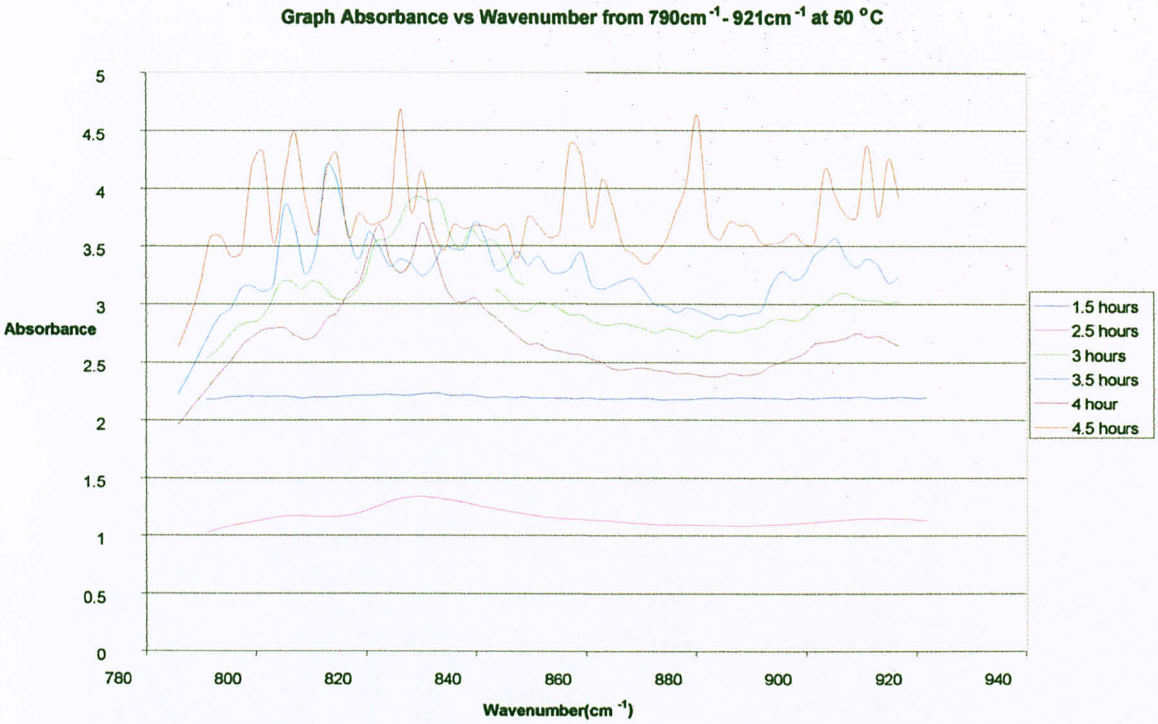



**Figure 20:** Graph for epoxy resin at 50°C at mid infrared

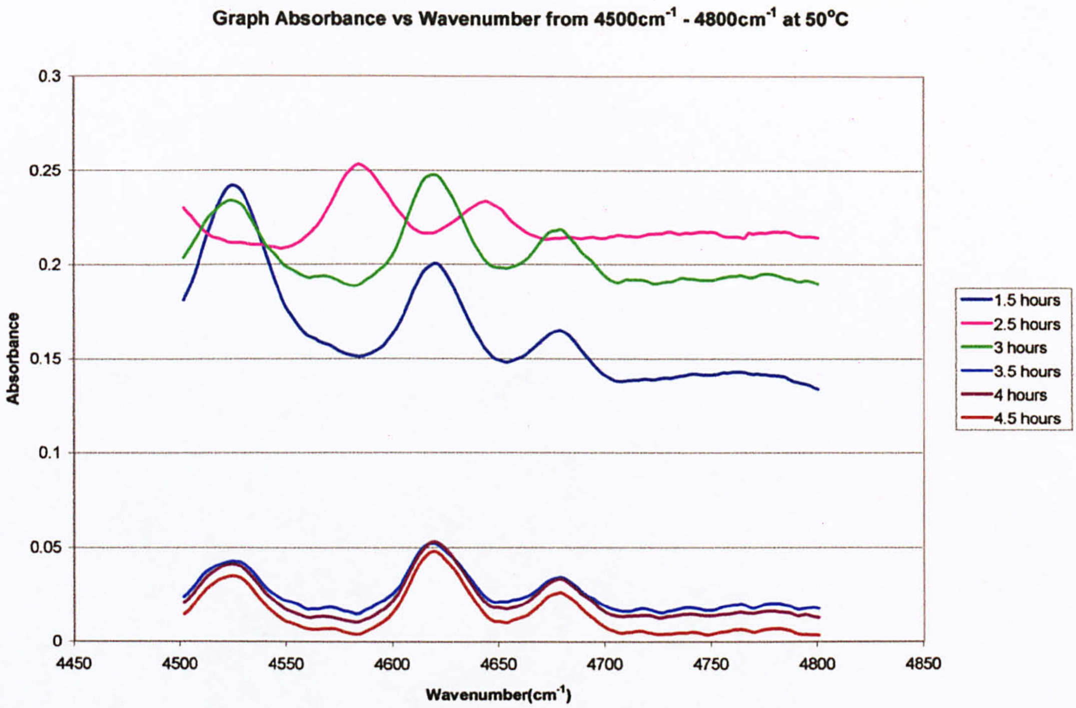



**Figure 21:** Graph for epoxy resin at 50°C at near infrared

### 4.1.1.3 Temperature at 120°C

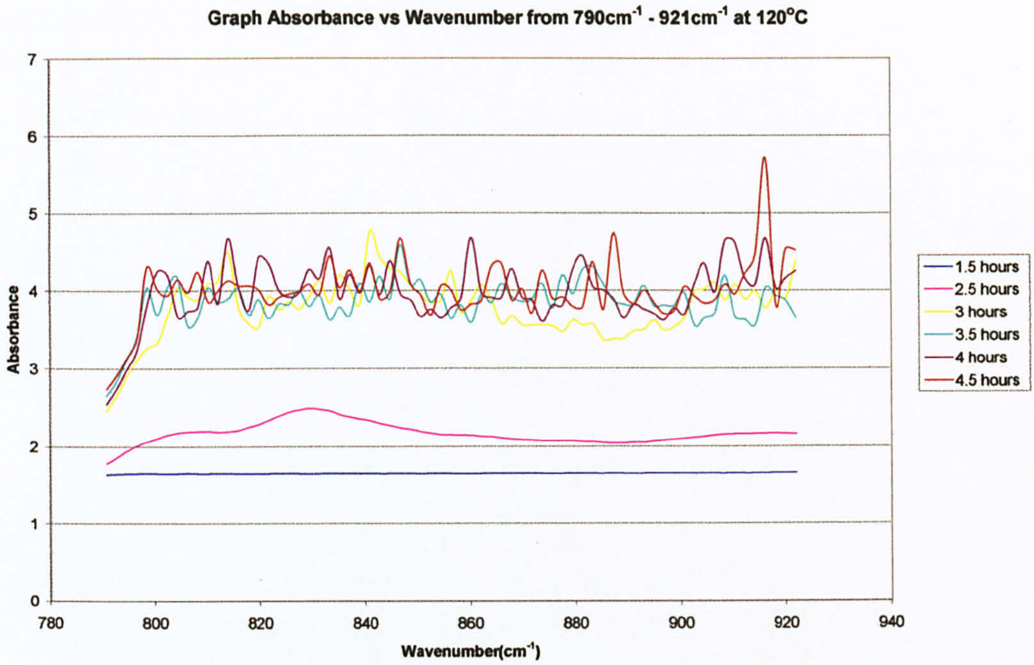



**Figure 22:** Graph for epoxy resin at 120°C at mid infrared

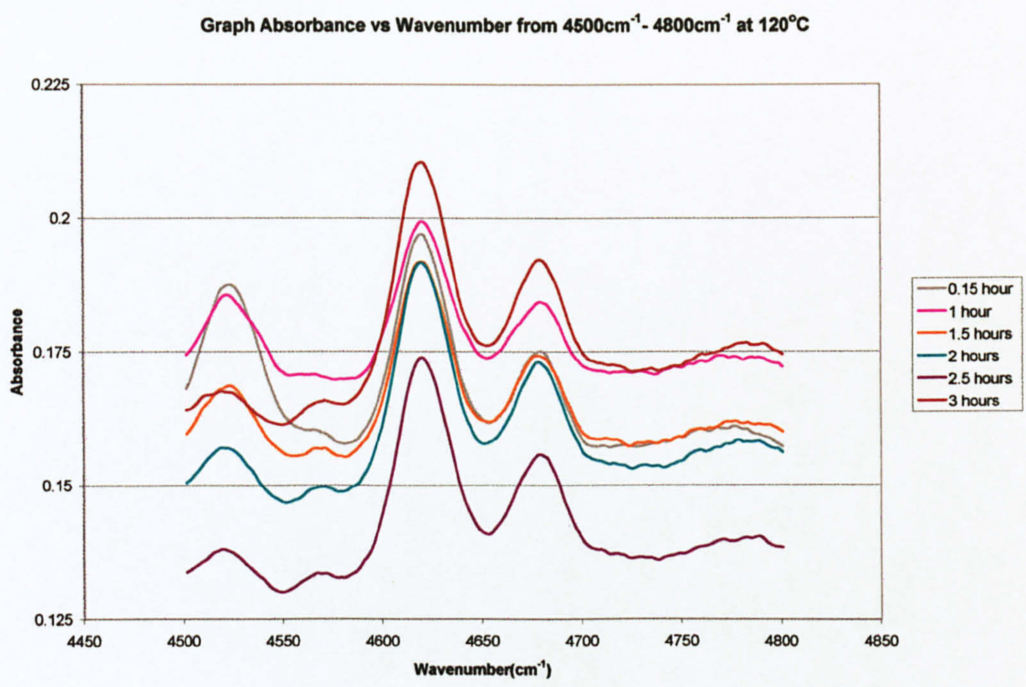



**Figure 23:** Graph for epoxy resin at 120°C at near infrared

## 4.1.2 Effect of Water on Epoxy Resin

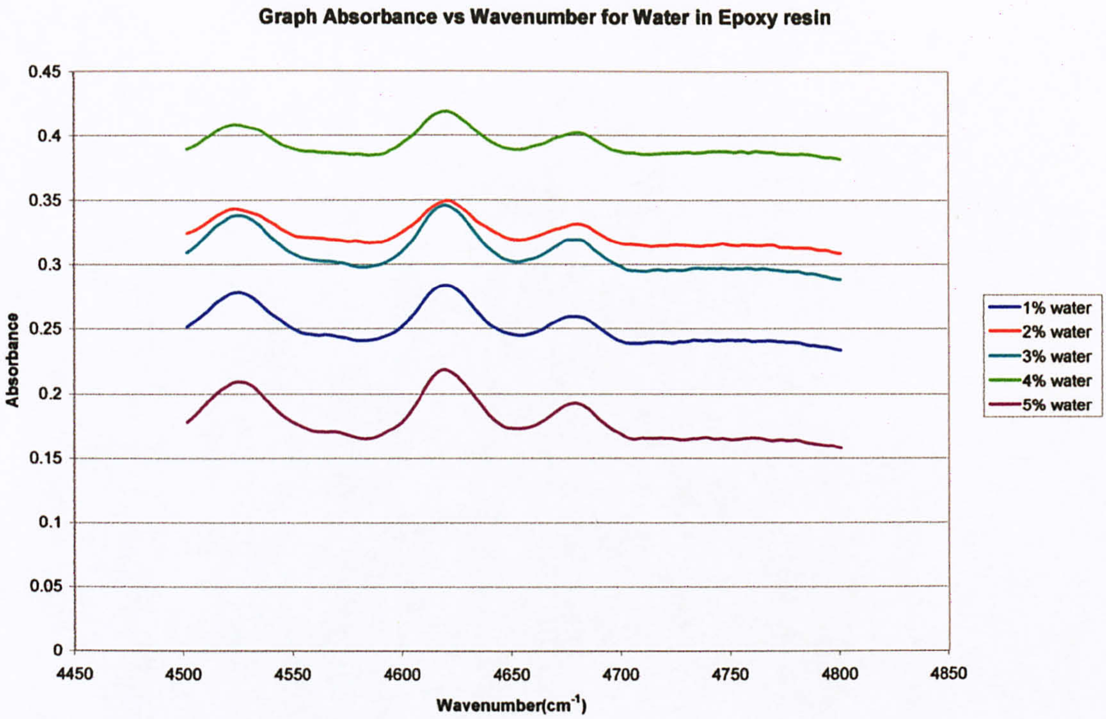



**Figure 24:** Graph for epoxy resin with water at near infrared

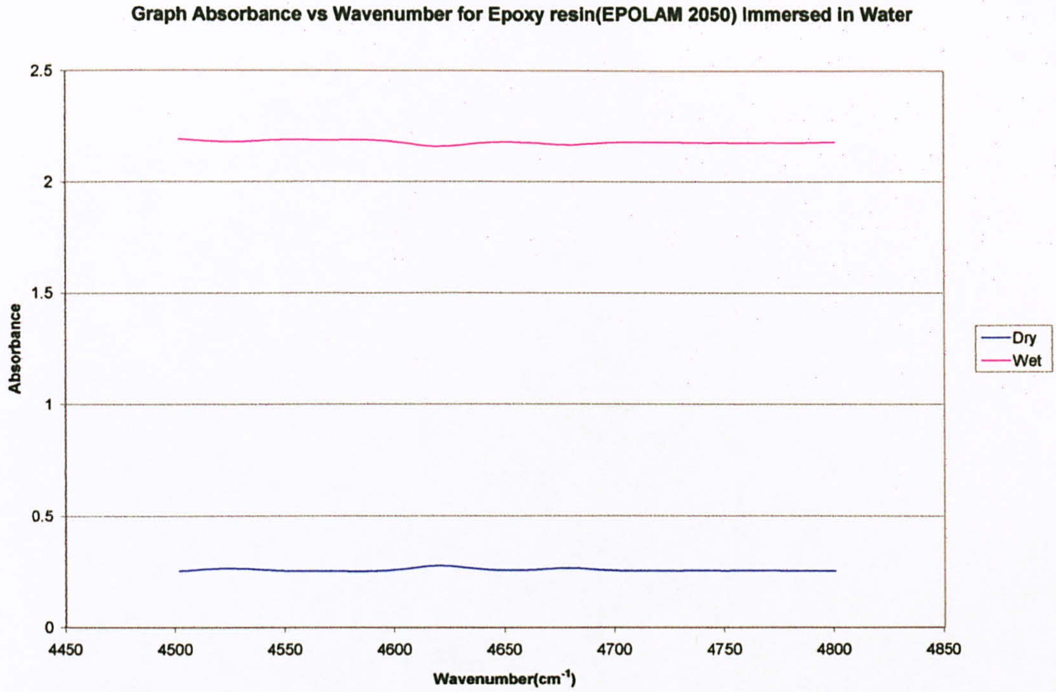



**Figure 25:** Graph for epoxy resin immersed in water at near infrared

## 4.2 Discussions

In this project, the epoxy resin and hardener used is EPOLAM 2050. It has good thermal resistance, good wetting on fabrics and filler, low shrinkage and easier curing cycle. Table 1 shows the physical properties for resin and hardener of EPOLAM 2050. [7]

**Table 3:** Physical Properties of EPOLAM 2050 resin and hardener

| Physical Properties | | | |
|---|---|---|---|
| **Composition** | **Resin** | **Hardener** | **Mixed** |
| Mix ratio by weight | 100 | 32 | - |
| Mix ratio by volume at 25°C | 100 | 40 | - |
| Colour | Clear green | Amber | Clear green |
| Viscosity at 25°C (MPa.s) | 4300 | 80 | 2000 |
| Specific gravity at 25°C | 1.19 | 0.96 | - |
| Pot life at 25°C on 264g (min) | - | - | 70-90 |

**Figure 26:** EPOLAM 2050 Epoxy Resin

**Figure 27:** EPOLAM 2050 Curing Agent

There are many ways to prepare the resin to be analyzed under infrared. Epoxy resin in thin film form is preferred. It is because the resin can be directly tested using infrared without sample preparation. Before the resin mixture is spread uniformly on the glass, the glass must be applied with a layer of wax. The step is important in order to easily cut the cured resin out from the glass.

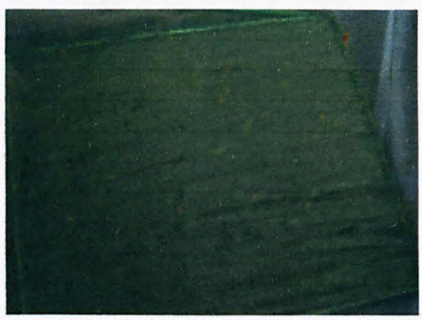


**Figure 28:** Application of Wax of the Glass

The mixing ratio of resin and hardener is also an important step. The value for mixing ratio is 10:3.2 by weight as indicated in Material Safety Data Sheet (MSDS). So when resin and hardener are used in larger amount, calculation needs to be performed. The step of calculation is shown below. When the weigh of resin is known, by applying this formula, weight of hardener can be calculated.

$$\frac{Weight\_of\_resin}{Weight\_of\_hardener} = \frac{10}{3.2}$$

$$Weight\_of\_resin = \frac{10}{3.2} x Weight\_of\_hardener$$

For infrared, there wavenumber is set from 400cm$^{-1}$ to 7800cm$^{-1}$. The purpose is to study polymerization of resin at both mid infrared (400cm$^{-1}$ to 4000cm$^{-1}$) and near infrared (4000cm$^{-1}$ to 12000cm$^{-1}$). Then the wavenumber that produce more accurate results will be chosen. In order to study on the polymerization process of epoxy resin and hardener, there are two functional groups to be observed. These functional groups are epoxide group and p-phenylene group. At 50°C, epoxy resin is still in the process of hardening on the first one and half hour, therefore infrared test could not be carried out. In order to compare the curing process of epoxy at different time, this formula can be applied.

$$Ratio\_of\_Height = \frac{Height\_of\_epoxide\_at\_time\_n}{Height\_of\_p-phenylene\_at\_time\_n}$$

From the graph absorbance versus wavenumber for epoxy resin, height of epoxide peak can be measure and compared to height of p-phenylene. Epoxide group is chosen because eventually the peak will reduce as the curing process goes on. Meanwhile, p-phenylene is selected because this peak will not be affected when the polymerization process takes place and remain constant throughout the reaction.[7] Then the ratio of height is compared for every 30 minutes time interval. Other than 50°C, the epoxy resin is also located in oven at 120°C.

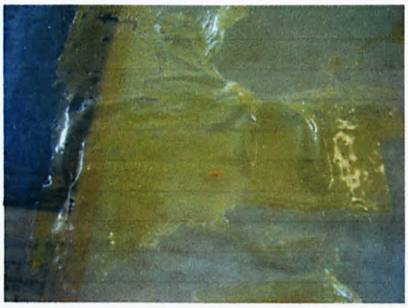


**Figure 29:** Epoxy Resin at Room Temperature

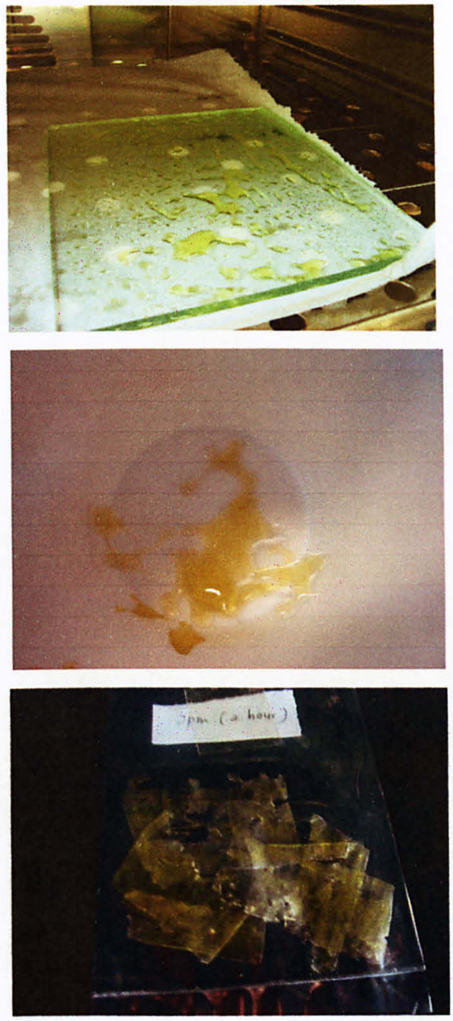


**Figure 30:** Resin at Elevated Temperature

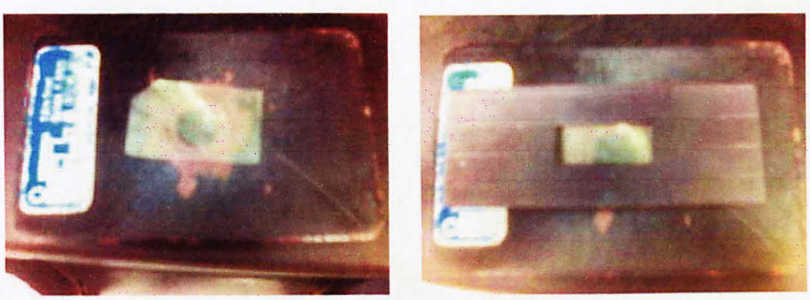
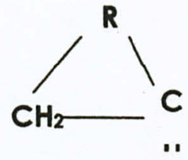
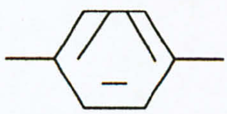


**Figure 31:** Preparation of Sample for Infrared Test

**Table 4:** Band assigment of chemical group from NIR and MIR absorption spectra

| Chemical Group | Chemical Structure | Observed Wavenumber |
|---|---|---|
| Epoxide |  | 914cm$^{-1}$ and 4526cm$^{-1}$ |
| p-phenylene |  | 795cm$^{-1}$ and 4673cm$^{-1}$ |

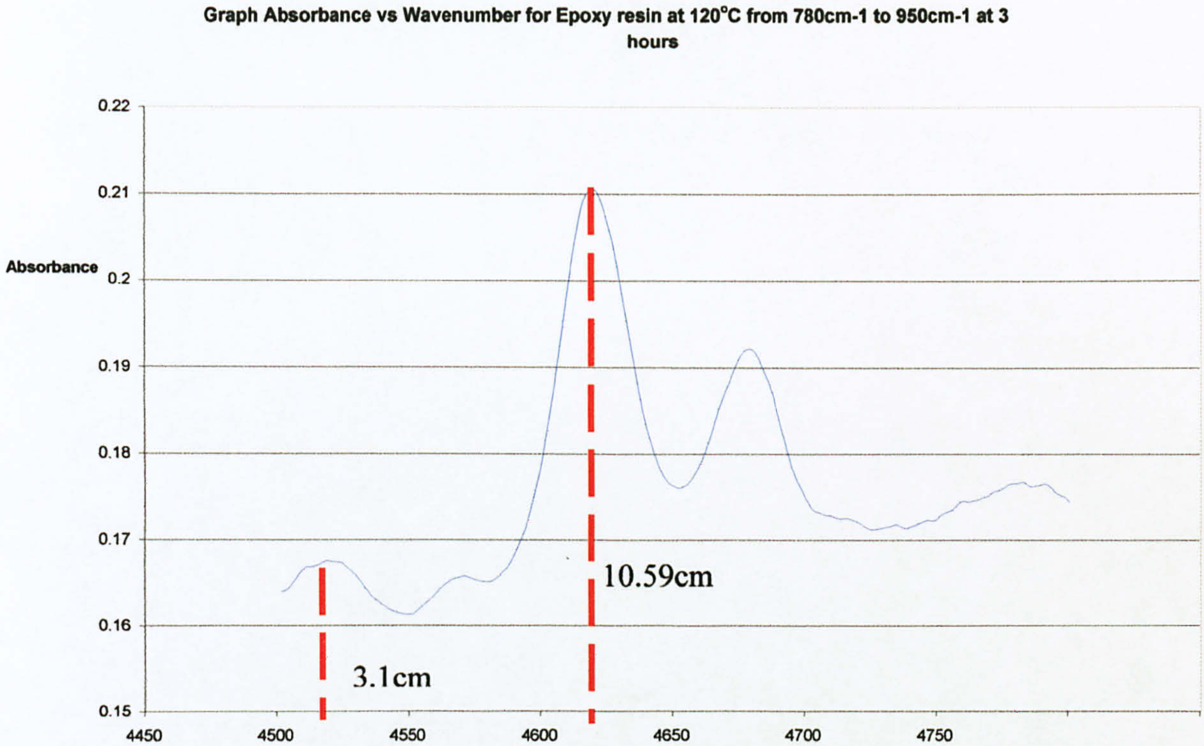



**Figure 32:** Graph absorbance vs wavelength for epoxy resin at 120°C at 3 hours (NIR)

Figure 8 shows the graph of absorbance versus wavelength for epoxy resin at 120°C for 3 hours. The wavenumber for epoxide at near infared(NIR) is 4526cm$^{-1}$ while for p-phenylene is 4620cm$^{-1}$. Ratio of height for both chemical groups is calculated below.

$$Ratio\_of\_height = \frac{3.1cm}{10.59cm} = 0.284$$

The summarization of height epoxy resin at different time interval is shown below.

**Table 5:** Calculation of height of peak for epoxy resin at 120°C at NIR

| Time interval(hour) | Height(cm) | | Ratio |
|---|---|---|---|
| | Epoxide | p-phenylene | |
| 0.5 | 8.80 | 11.10 | 0.79 |
| 1.0 | 6.10 | 10.15 | 0.60 |
| 1.5 | 3.30 | 10.20 | 0.32 |
| 2.0 | 3.60 | 11.30 | 0.32 |
| 2.5 | 2.75 | 11.75 | 0.23 |
| 3.0 | 3.10 | 10.59 | 0.29 |

At mid infrared (MIR), the wavenumber for epoxide is 914cm$^{-1}$ while p-phenylene will be 798cm$^{-1}$. The same formula is applicable in this graph to in order to calculate the height of respective peak.

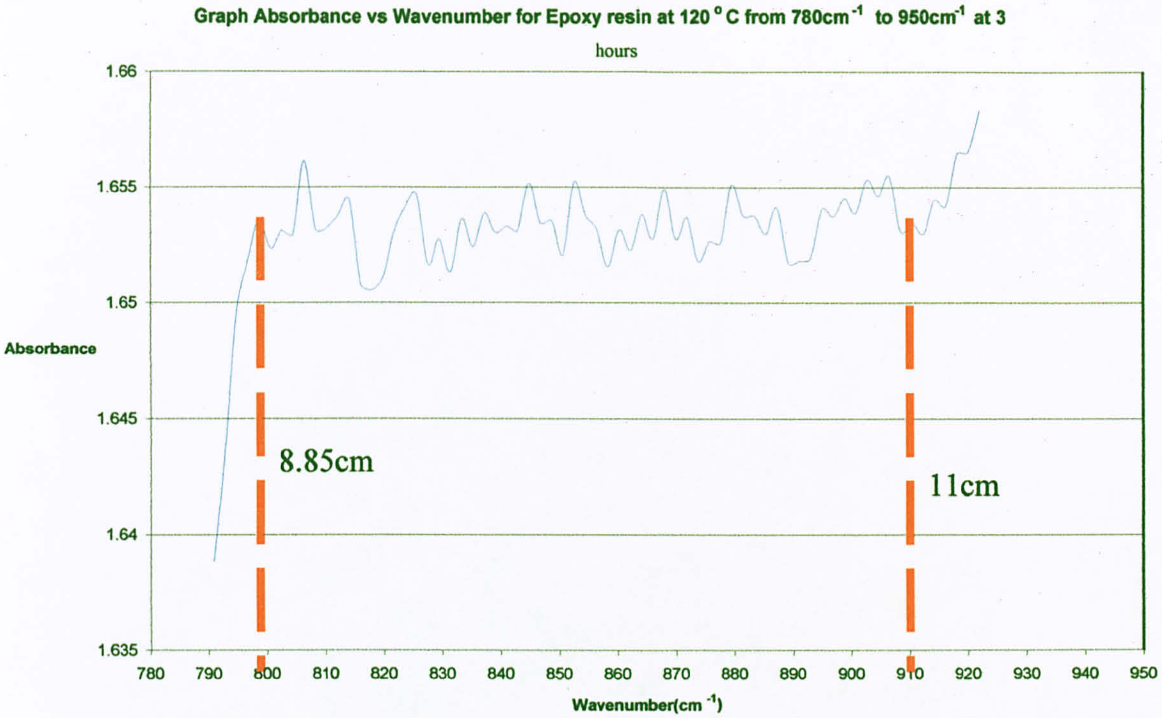



**Figure 33:** Graph absorbance vs wavelength for epoxy resin at 120°C at 3 hours (MIR)

**Table 6:** Calculation of height of peak for epoxy resin at 120°C at MIR

| Time interval(hour) | Height(cm) | | Ratio |
|---|---|---|---|
| | Epoxide | p-phenylene | |
| 0.5 | 9.65 | 9.25 | 1.04 |
| 1.0 | 7.90 | 6.00 | 1.32 |
| 1.5 | 7.85 | 5.00 | 1.57 |
| 2.0 | 7.30 | 7.30 | 1.00 |
| 2.5 | 10.85 | 6.20 | 1.75 |
| 3.0 | 11.00 | 8.85 | 1.24 |

From both tables, it could be concluded that near infared will give better results compared to mid infrared. The ratio will decrease as the time goes on, it indicated the curing process is taking place where epoxide group will decrease.

**Table 7:** Calculation of height of peak for epoxy resin at 50°C at NIR

| Time interval(hour) | Height(cm) | | Ratio |
|---|---|---|---|
| | Epoxide | p-phenylene | |
| 1.5 | 12.6 | 8.90 | 1.42 |
| 2.5 | 2.60 | 4.80 | 0.54 |
| 3.0 | 10.65 | 12.90 | 0.83 |
| 3.5 | 10.90 | 14.20 | 0.77 |
| 4.0 | 9.10 | 11.7 | 0.76 |
| 4.5 | 7.75 | 10.60 | 0.73 |

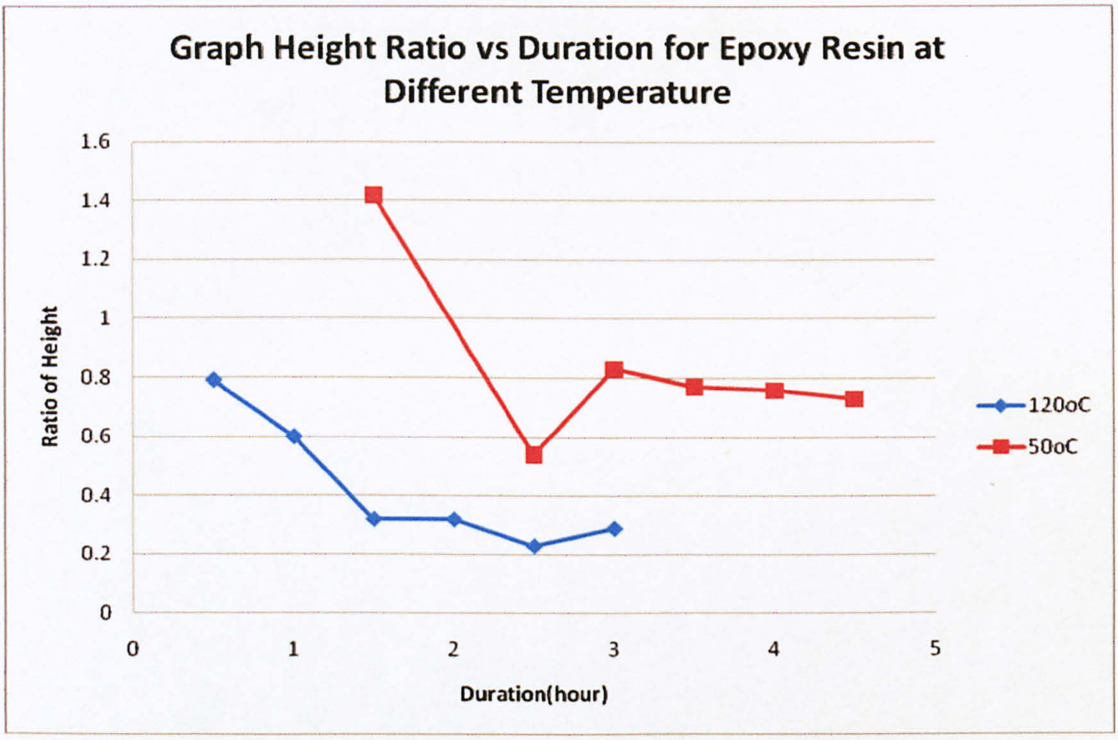



**Figure 34:** Graph Height Ratio vs Duration for Epoxy Resin at 120°C and 50°C

At 50°C, the same steps are applied to obtain the ratio for peak of height for epoxy resin at every time interval. At this temperature, the resin will only start hardens at 1.5 hours. Only at near infrared the height of peak could be measured, it is because at mid infrared, the peak is not stable and uneven, and led to difficulties in measuring the height. Before 1.5 hours, the resin is still in gel form and test could not be run. Unlike at higher temperature, the resin will harden faster. Therefore, it can be said that the temperature will give big impact on polymerization process of resin. At higher temperature, polymerization process will occurs faster. It is because the epoxy resin and amines molecules will move and react faster when they obtain energy from the heat.

At room temperature which is about 24°C, the ratio of height is shown on below Table 8. As the curing process goes on, the ratio of height will decrease until it achieves stable ratio. The polymerization process might be completed and no further crosslinking occurs.

**Table 8:** Calculation of height of peak for epoxy resin at room temperature

| Duration | Height(cm) | | Ratio |
|---|---|---|---|
| (Day) | Epoxide | p-phenylene | |
| 1 | 9.30 | 6.30 | 1.48 |
| 2 | 7.20 | 6.30 | 1.14 |
| 3 | 7.50 | 7.10 | 1.06 |
| 4 | 6.90 | 8.00 | 0.86 |
| 5 | 5.90 | 7.90 | 0.75 |
| 6 | 6.00 | 8.50 | 0.71 |
| 7 | 6.10 | 8.70 | 0.70 |

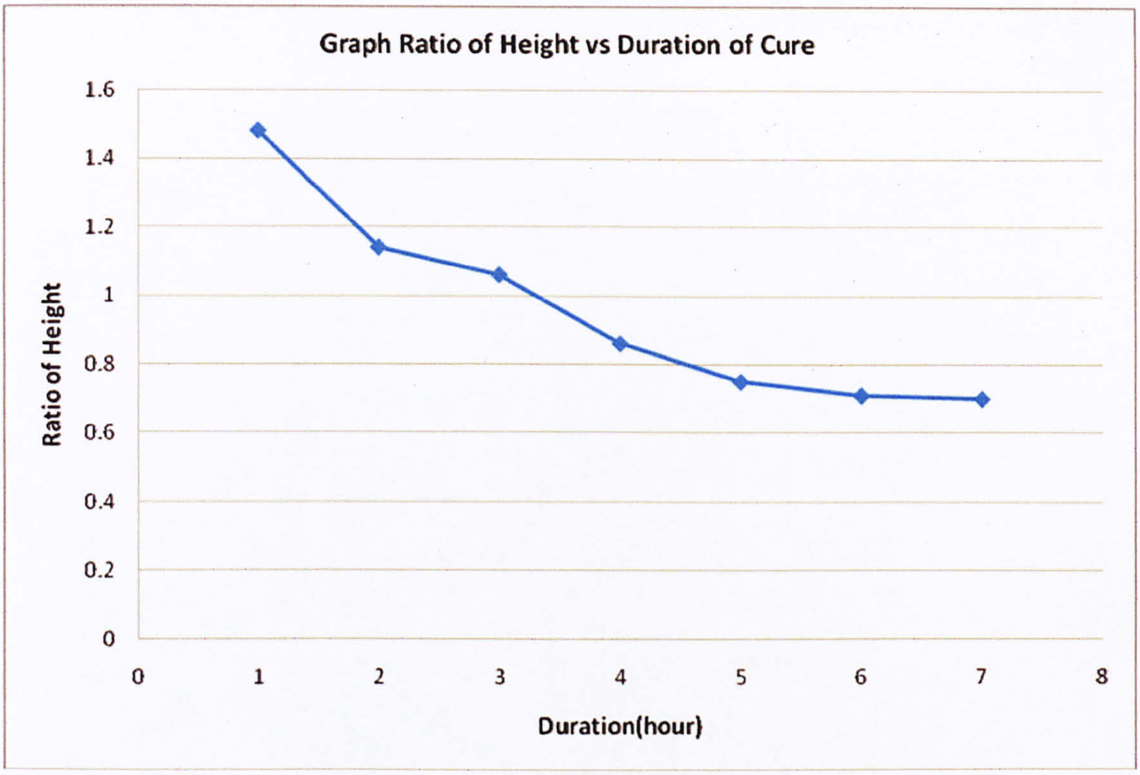



**Figure 35:** Graph Height Ratio vs Duration of Cure

For observing the effect of water on epoxy resin, water is added during the mixing of epoxy resin and hardener. This step is done to know the reaction of water with resin and hardener. The amount of water measured is based on the total weight of the resin and hardener.

$$\%\_of\_water = \frac{Weight\_of\_water}{Total\_weight\_of\_resin\_and\_hardener} x100\%$$

The amount of water is varied from 1% to 5%. Excessive amount of water will limit the movement of water molecules and forces the water to form cluster with other water molecules.

Since NIR peak is easier to be measured, therefore the following results will be in NIR. Table 7 is the calculation for peak height of epoxy resin with different amount of water. From the ratio of height, it can be said that as the amount of water in epoxy resin increases, crosslinking will take place at higher rate. It is because the hydroxide, OH group in water molecules will form hydrogen bond with epoxide group in epoxy resin. [8]

**Table 9:** Calculation of height of peak for epoxy resin with water at NIR

| Amount of water (%) | Height(cm) | | Ratio |
|---|---|---|---|
| | Epoxide | p-phenylene | |
| 1 | 10.5 | 6.00 | 1.75 |
| 2 | 10.1 | 7.00 | 1.44 |
| 3 | 10.95 | 7.50 | 1.46 |
| 4 | 8.45 | 5.75 | 1.47 |
| 5 | 11.15 | 8.10 | 1.38 |

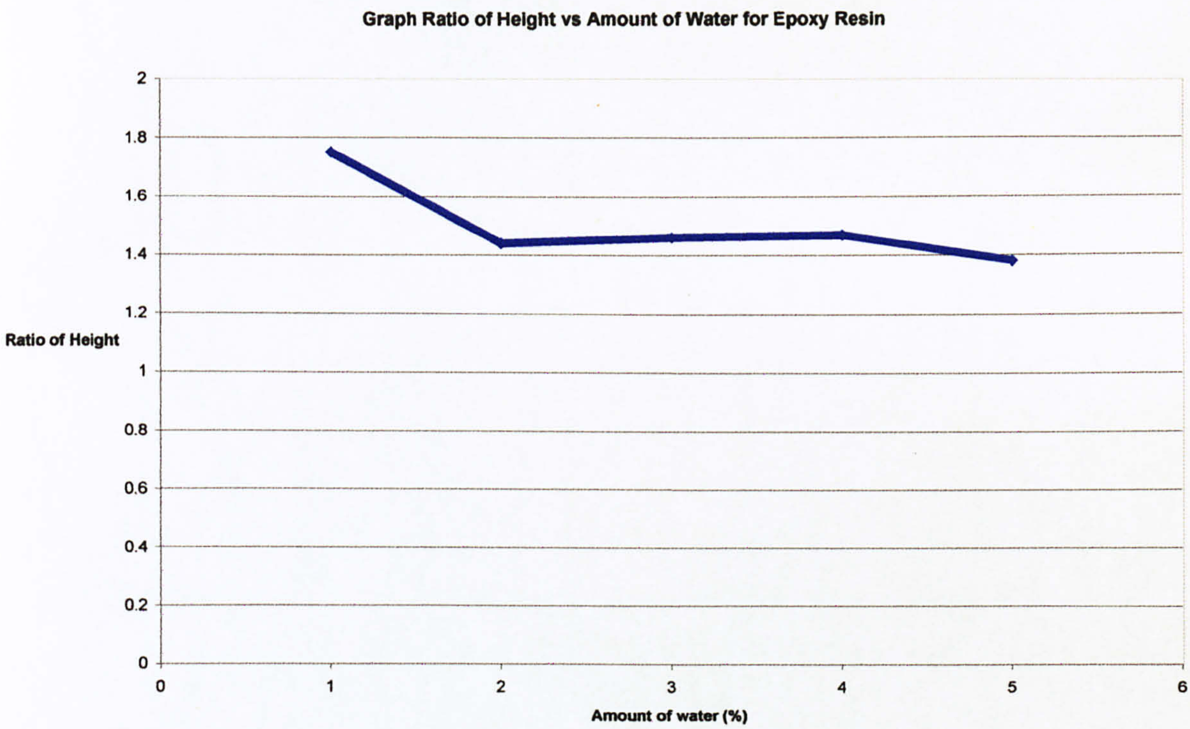



**Figure 36:** Graph ratio of height for epoxy resin with different amount of water

This phenomenon also applied on immersing the epoxy plate in water after mixing process. When there are a lot of water molecules, the epoxy's colour will change immediately from a light transparent green to white colour. The results is illustrates in Table 8 below. It is obvious that at wet environment, the curing process in epoxy resin will occur faster compared to at dry condition (room temperature).

**Table 10:** Calculation of height of peak for epoxy resin with different amount of water at NIR

| Time interval(hour) | Height(cm) | | Ratio |
|---|---|---|---|
| | Epoxide | p-phenylene | |
| Dry | 6.35 | 7.10 | 0.89 |
| Wet | 5.00 | 9.15 | 0.55 |

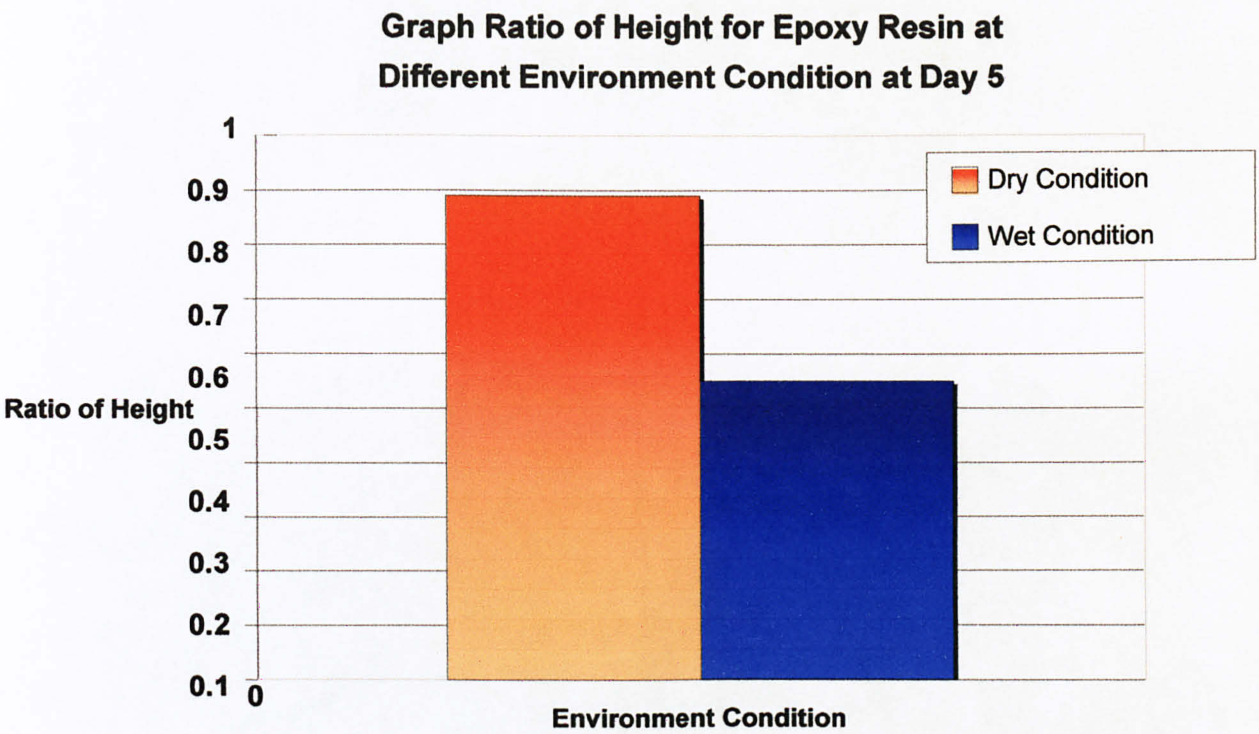



**Figure 37:** Graph Height Ratio for Resin at Different Environment

# CHAPTER 5

# CONCLUSIONS AND RECOMMENDATIONS

## 5.1 Conclusions

From this research, it improves the author understanding on epoxy resin chemically. The objectives of this researched are achieved which is to determine the curing characteristic of epoxy resin at various conditions. At elevated temperature, the resin and hardener will react faster as the molecules gain more kinetic energy to move. At watery condition, the epoxy resin will cure faster too compared to the resin at dry condition. With this information, the curing of epoxy resin at different conditions can be roughly known before applying on technical structural.

## 5.2 Recommendations

As recommendations, this research can be expanded by using various types of mechanical and spectroscopy analysis which are:

a) Raman spectroscopy
b) Differential Scanning Calorimeter test
c) Tensile test

With these tools, the properties of epoxy can be studied more deeply. As mentioned in literature review, Raman spectroscopy can be used to support the results obtained from infrared analysis. Both spectroscopy methods have almost the same function eventhough the theory and techniques are different. Meanwhile, by applying Differential Scanning Calorimeter, the percentage of curing for resin can be calculated together with the information on glass transition temperature. The information on changes in curing reaction can be more complete too with mechanical test.

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8. F.X Perrin, Minh Hanh Nguyen, J.L. Vernet, Water Transport in Epoxy-Aliphatic Amine Networks-Influence of Curing Cycles

# APPENDIX MATERIAL SAFETY DATA SHEET

AXSON TECHNOLOGIES

# EPOLAM 2050

## LAMINATING SYSTEM FOR TOOLING

*Tg 125°C*

## DESCRIPTION

*Production of tooling by laminating. Used in concrete or in casting by addition of fillers.*

## PROPERTIES

- *Product free of MDA*
- *Good thermal resistance*
- *Good wetting of fabrics and fillers*
- *Good resistance in a vertically side*
- *Low exothermy*
- *Low shrinkage*
- *Easier curing cycle*

| PHYSICAL PROPERTIES | | | | |
|---|---|---|---|---|
| Composition | | **RESIN** | **HARDENER** | **MIXED** |
| Mix ratio by weight<br>Mix ratio by volume at 25°C | | 100<br>100 | 32<br>40 | |
| Aspect | | liquid | liquid | liquid |
| Colour | | clear green | amber | clear green |
| Viscosity at 25°C (mPa.s) | BROOKFIELD LVT | 4,300 | 80 | 2,000 |
| Specific gravity at 25°C<br>Specific gravity of cured product at 23°C | ISO 1675 : 1985<br>ISO 2781 : 1996 | 1.19<br>- | 0.96<br>- | -<br>1.12 |
| Pot life at 25°C on 264 g (min) | Gel Timer TECAM | | | 70 - 90 |

| MECHANICAL PROPERTIES at 23°C (1) | | | |
|---|---|---|---|
| Flexural modulus | ISO 178 :2001 | MPa | 3,300 |
| Flexural strength | ISO 178 :2001 | MPa | 105 |
| Tensile strength | ISO 527 :1993 | MPa | 45 |
| Compressive strength | ISO 604 :2002 | MPa | 110 |
| Final hardness | ISO 868 :2003 | Shore D | 87 |
| Glass temperature transition | ISO 11359 : 2002 | °C | 125 |
| Demoulding time at room temperature | | hr | 24 - 36 |
| Complete hardening time at room temperature | | d | 5 |

*(1) : Average values obtained on standard specimens after 15 hours at 120°C curing*



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Axson TECHNOLOGIES

# EPOLAM 2050

**LAMINATING SYSTEM FOR TOOLING**
***Tg 125°C***

## PROCESSING CONDITIONS

***LAMINATING:*** *After mixing according to the indicated ratio, impregnate the reinforcements by the different laminating processes. Demould after 24 hours polymerisation at room temperature. Then cure (see § thermal treatment).*

***CONCRETE:*** *In order to produce an aluminium concrete, use 120 g to 150 g of EPOLAM 2050 mix for 500 g of RZ 1019 granulate + 500 g of RZ 1020 granulate. Mix the whole together with a planetary agitator and tamp down the concrete on to the laminate*

## THERMAL TREATMENT

*A good rate of polymerisation is obtained at room temperature. A curing is not necessary when using a tooling at a temperature under 50°C.*

*On the other hand a thermal treatment is necessary for applications requiring a good thermal resistance until 125°C. This is the reason why the tooling or the part must be cured for 8 to 12 hours at an inferior temperature of 20°C to 30°C than its working temperature. Then the processing of the tooling will allow to finish the thermal treatment.*

***Caution:*** *When curing tooling of high dimension it is necessary to use suitable supports in order to avoid any risk of distortion.*

*Laminating thickness must not exceed 12 mm in only one operation.*

## HANDLING PRECAUTIONS

*Normal health and safety precautions should be observed when handling these products :*
*Ensure good ventilation*
*Wear gloves, safety glasses and waterproof clothes.*
*For further information, please consult the product safety data sheet.*

## STORAGE CONDITIONS

*Shelf life is 12 months for in a dry place and in original unopened containers at a temperature between 15 and 25° C. Any open can must be tightly closed under dry nitrogen blanket.*

## PACKAGING

| ***RESIN*** | ***HARDENER*** |
|---|---|
| *1 x 20 kg* | *1 x 6,40 kg* |
| *1 x 50 kg* | *1 x 16 kg* |

## GUARANTEE

*The information of our technical data sheet are based on our present knowledge and the result of tests conducted under precise conditions. It is the responsibility of the user to determine the suitability of AXSON products, under their own conditions before commencing with the proposed application. AXSON refuse any guarantee about the compatibility of a product with any particular application. AXSON disclaim all responsibility for damage from any incident which results from the use of these products. The guarantee conditions are regulated by our general sale conditions.*

## 1 Identification of the substance/preparation and of the company/undertaking

· **Product details**

· **Trade name: EPOLAM 2050 RESINE**
· **Application of the substance / the preparation** Epoxy resin

· **Manufacturer/Supplier:**
AXSON FRANCE
Rue de l'Equerre
F-95310 SAINT OUEN L'AUMONE
Tél.(33-1)34 40 34 60

· **Further information obtainable from:** LAB. R&D -(33) 01 34 40 34 60
· **Information in case of emergency:** ORFILA : (33)01 45 42 59 59

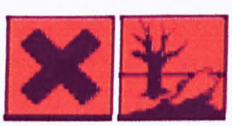
## 2 Composition/information on ingredients

· **Chemical characterization**
· **Description:** Mixture of substances listed below with nonhazardous additions.

| · **Dangerous components:** | | |
|---|---|---|
| CAS: 25068-38-6<br>EINECS: 500-033-5 | reaction product: bisphenol A-(epichlorhydrin); epoxy resin (number average molecular weight ≤ 700)<br>Xi, N; R 36/38-43-51/53 | 25-50% |
| CAS: 28768-32-3 | Tetraglycidylether methylene bis aniline<br>Xn, N; R 21/22-40-43-51/53 | 2.5-10% |

## 3 Hazards identification

· **Hazard description:**



Xn Harmful
N Dangerous for the environment

· **Information concerning particular hazards for human and environment:**
R 36/38 Irritating to eyes and skin.
R 40 Limited evidence of a carcinogenic effect.
R 43 May cause sensitisation by skin contact.
R 51/53 Toxic to aquatic organisms, may cause long-term adverse effects in the aquatic environment.
Contains epoxy constituents. See information supplied by the manufacturer.

## 4 First-aid measures

· **After inhalation:**
Supply fresh air and to be sure call for a doctor.
In case of unconsciousness place patient stably in side position for transportation.
· **After skin contact:** Immediately wash with water and soap and rinse thoroughly.
· **After eye contact:**
Rinse opened eye for several minutes under running water. Then consult a doctor.
· **After swallowing:** Do not induce vomiting; call for medical help immediately.



(Contd. on page 2)

***Trade name: EPOLAM 2050 RESINE***

(Contd. of page 1)

## 5 Fire-fighting measures

· ***Suitable extinguishing agents:***
*CO2, powder or water spray. Fight larger fires with water spray or alcohol resistant foam.*
· ***For safety reasons unsuitable extinguishing agents:*** *Water with full jet*
· ***Special hazards caused by the substance, its products of combustion or resulting gases:***
*In case of fire, the following can be released:*
*Carbon monoxide (CO)*
*Nitrogen oxides (NOx)*
· ***Protective equipment:***
*Wear fully protective suit.*
*Wear self-contained respiratory protective device.*
· ***Additional information***
*Dispose of fire debris and contaminated fire fighting water in accordance with official regulations.*

## 6 Accidental release measures

· ***Person-related safety precautions:*** *Wear protective equipment. Keep unprotected persons away.*
· ***Measures for environmental protection:***
*Do not allow product to reach sewage system or any water course.*
· ***Measures for cleaning/collecting:***
*Absorb with liquid-binding material (sand, diatomite, acid binders, universal binders, sawdust).*
· ***Additional information:*** *See Section 8 for information on personal protection equipment.*

## 7 Handling and storage

· ***Handling:***
· ***Information for safe handling:*** *Ensure good ventilation/exhaustion at the workplace.*

· ***Storage:***
· ***Requirements to be met by storerooms and receptacles:*** *Prevent any seepage into the ground.*
· ***Information about storage in one common storage facility:*** *Store away from foodstuffs.*
· ***Further information about storage conditions:***
*Store in cool, dry conditions in well sealed receptacles.*
· ***Storage class:***
· ***Class according to regulation on flammable liquids:*** *Void*

## 8 Exposure controls/personal protection

· ***Ingredients with limit values that require monitoring at the workplace:***
*The product does not contain any relevant quantities of materials with critical values that have to be monitored at the workplace.*

· ***Personal protective equipment:***
· ***General protective and hygienic measures:***
*The usual precautionary measures are to be adhered to when handling chemicals.*
*Immediately remove all soiled and contaminated clothing*
*Wash hands before breaks and at the end of work.*
*Avoid contact with the eyes and skin.*
· ***Respiratory protection:*** *Not necessary if room is well-ventilated.*

(Contd. on page 3)

(Contd. of page 2)

· ***Protection of hands:***

*Protective gloves*

· ***Material of gloves*** *PVC gloves*
· ***Eye protection:***

*Safety glasses*

*Tightly sealed goggles*
· ***Body protection:*** *Protective work clothing*

## *9 Physical and chemical properties*

· ***General Information***

| | |
|---|---|
| ***Form:*** | *Fluid* |
| ***Colour:*** | *Green* |
| ***Odour:*** | *Characteristic* |
| · ***Change in condition*** | |
| ***Melting point/Melting range:*** | *NA°C* |
| ***Boiling point/Boiling range:*** | *>200°C (DIN 53171)* |
| · ***Flash point:*** | *>110°C (ISO 2719)* |
| · ***Ignition temperature:*** | *>300°C (DIN 51 794)* |
| · ***Decomposition temperature:*** | *>200°C (DIN 53171)* |
| · ***Self-igniting:*** | *Product is not selfigniting.* |
| · ***Danger of explosion:*** | *Product does not present an explosion hazard.* |
| · ***Density at 20°C:*** | *1.19 g/cm³* |
| · ***Solubility in / Miscibility with water:*** | *Insoluble.* |
| · ***organic solvents:*** | *Soluble in many organic solvents.* |

## *10 Stability and reactivity*

· ***Thermal decomposition / conditions to be avoided:***
*No decomposition if used and stored according to specifications.*
· ***Dangerous reactions***
*May produce violent reactions with bases and numerous organic substances including alcohols and amines.*
*Exothermic polymerization.*
· ***Dangerous decomposition products:*** *Irritant gases/vapours*



(Contd. on page 4)

(Contd. of page 3)

## *11 Toxicological information*

· ***Acute toxicity:***

| · ***LD/LC50 values relevant for classification:*** | | |
|---|---|---|
| ***25068-38-6 reaction product: bisphenol A-(epichlorhydrin); epoxy resin (number average molecular weight ≤ 700)*** | | |
| *Oral* | *LD50* | *11400 mg/kg (rat)* |
| *Dermal* | *LD50* | *>2000 mg/kg (rabbit)* |

· ***Primary irritant effect:***
· ***on the skin:*** *Irritant to skin and mucous membranes.*
· ***on the eye:*** *Irritating effect.*
· ***Sensitization:*** *Sensitization possible through skin contact.*

## *12 Ecological information*

· ***Ecotoxical effects:***

| · ***Acquatic toxicity:*** | |
|---|---|
| ***28768-32-3 Tetraglycidylether methylene bis aniline*** | |
| *LC 50 (96h)* | *7 mg / l (fish)* |

## *13 Disposal considerations*

· ***Product:***
· ***Recommendation***
*Must not be disposed together with household garbage. Do not allow product to reach sewage system.*
*Dispose of the product by burning in a suitable incinerator or bury in an approved landfield following all applicable local and/or national regulations.*

| · ***European waste catalogue*** | |
|---|---|
| *20 01 27* | *paint, inks, adhesives and resins containing dangerous substances* |

· ***Uncleaned packaging:***
· ***Recommendation:***
*Empty containers may not be disposed of unless any remaining material adhering to the internal walls has been removed.*
*Disposal must be made according to official regulations.*

## *14 Transport information*

· ***Land transport ADR/RID (cross-border)***

· ***ADR/RID class:*** *9 Miscellaneous dangerous substances and articles.*
· ***Danger code (Kemler):*** *90*
· ***UN-Number:*** *3082*
· ***Packaging group:*** *III*
· ***Hazard label*** *9*

(Contd. on page 5)

(Contd. of page 4)

· ***Description of goods:*** *3082 ENVIRONMENTALLY HAZARDOUS SUBSTANCE, LIQUID, N.O.S. (epoxy resins)*

· ***Maritime transport IMDG:***

· ***IMDG Class:*** *9*
· ***UN Number:*** *3082*
· ***Label*** *9*
· ***Packaging group:*** *III*
· ***Marine pollutant:*** *No*
· ***Proper shipping name:*** *ENVIRONMENTALLY HAZARDOUS SUBSTANCE, LIQUID, N.O.S. (epoxy resins)*

· ***Air transport ICAO-TI and IATA-DGR:***

· ***ICAO/IATA Class:*** *9*
· ***UN/ID Number:*** *3082*
· ***Label*** *9*
· ***Packaging group:*** *III*
· ***Proper shipping name:*** *ENVIRONMENTALLY HAZARDOUS SUBSTANCE, LIQUID, N.O.S. (epoxy resins)*

## 15 Regulatory information

· ***Labelling according to EU guidelines:***
*The product has been marked in accordance with EU Directives / respective national laws.*

· ***Code letter and hazard designation of product:***
*Xn Harmful*
*N Dangerous for the environment*

· ***Hazard-determining components of labelling:***
*reaction product: bisphenol A-(epichlorhydrin); epoxy resin (number average molecular weight ≤ 700)*
*Tetraglycidylether methylene bis aniline*

· ***Risk phrases:***
*36/38 Irritating to eyes and skin.*
*40 Limited evidence of a carcinogenic effect.*
*43 May cause sensitisation by skin contact.*
*51/53 Toxic to aquatic organisms, may cause long-term adverse effects in the aquatic environment.*

· ***Safety phrases:***
*26 In case of contact with eyes, rinse immediately with plenty of water and seek medical advice.*
*28 After contact with skin, wash immediately with plenty of water.*
*36/37/39 Wear suitable protective clothing, gloves and eye/face protection.*
*57 Use appropriate container to avoid environmental contamination.*
*60 This material and its container must be disposed of as hazardous waste.*

· ***Special labelling of certain preparations:***
*Contains epoxy constituents. See information supplied by the manufacturer.*

(Contd. of page 5)

· ***National regulations:***

· ***Classification according to VbF:*** *Void*

· ***Waterhazard class:*** *Water hazard class 2 (Self-assessment): hazardous for water.*

## 16 Other information

*This information is based on our present knowledge. However, this shall not constitute a guarantee for any specific product features and shall not establish a legally valid contractual relationship.*

· ***Relevant R-phrases***

*21/22 Harmful in contact with skin and if swallowed.*
*36/38 Irritating to eyes and skin.*
*40 Limited evidence of a carcinogenic effect.*
*43 May cause sensitisation by skin contact.*
*51/53 Toxic to aquatic organisms, may cause long-term adverse effects in the aquatic environment.*

· ***Department issuing MSDS:*** *Product Safety and Toxicology*

# Material Safety Data Sheet
**According to 91/155 EC**

Printing date 25.11.2002

Reviewed on 21.11.2002

## 1 Identification of the substance/preparation and of the company/undertaking

· **Product details**

· **Trade name: EPOLAM 2050 DURCISSEUR**

· **Application of the substance / the preparation** Epoxy curing agent

· **Manufacturer/Supplier:**
AXSON FRANCE
Rue de l'Equerre
F-95310 SAINT OUEN L'AUMONE
Tél.(33-1)34 40 34 60

· **Further information obtainable from:** LAB. R&D -(33) 01 34 40 34 60
· **Information in case of emergency:** ORFILA : (33)01 45 42 59 59

## 2 Composition/information on ingredients

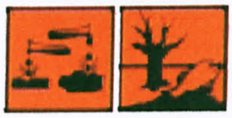
· **Chemical characterization**
· **Description:** Mixture of substances listed below with nonhazardous additions.

· **Dangerous components:**

| | | |
|---|---|---|
| CAS: 2855-13-2<br>EINECS: 220-666-8 | 3-aminomethyl-3,5,5-trimethylcyclohexylamine<br>C; R 21/22-34-43-52/53 | 50-100% |
| CAS: 39423-51-3<br>EINECS: 500-105-6 | Polyoxyalkyleneamine<br>C; R 21/22-34 | 10-25% |
| CAS: 68479-98-1<br>EINECS: 270-877-4 | diethylmethylbenzenediamine<br>Xn, N; R 21/22-36-48/22-50/53 | 10-25% |

## 3 Hazards identification

· **Hazard description:**



C Corrosive
N Dangerous for the environment

· **Information concerning particular hazards for human and environment:**
R 21/22 Harmful in contact with skin and if swallowed.
R 34 Causes burns.
R 43 May cause sensitisation by skin contact.
R 48/22 Harmful: danger of serious damage to health by prolonged exposure if swallowed.
R 51/53 Toxic to aquatic organisms, may cause long-term adverse effects in the aquatic environment.

## 4 First-aid measures

· **General information:** Immediately remove any clothing soiled by the product.
· **After inhalation:**
Supply fresh air; consult doctor in case of complaints.
In case of unconsciousness place patient stably in side position for transportation.
· **After skin contact:**
Immediately wash with water and soap and rinse thoroughly.
If skin irritation continues, consult a doctor.
· **After eye contact:**
Rinse opened eye for several minutes under running water. Then consult a doctor.

(Contd. on page 2)

(Contd. of page 1)

· **After swallowing:** Do not induce vomiting; call for medical help immediately.

## 5 Fire-fighting measures

· **Suitable extinguishing agents:**
$CO_2$, powder or water spray. Fight larger fires with water spray or alcohol resistant foam.
· **For safety reasons unsuitable extinguishing agents:** Water with full jet
· **Special hazards caused by the substance, its products of combustion or resulting gases:**
In case of fire, the following can be released:
Nitrogen oxides (NOx)
Carbon monoxide (CO)
· **Protective equipment:**
Wear self-contained respiratory protective device.
Wear fully protective suit.
· **Additional information**
Collect contaminated fire fighting water separately. It must not enter the sewage system.
Dispose of fire debris and contaminated fire fighting water in accordance with official regulations.

## 6 Accidental release measures

· **Person-related safety precautions:** Wear protective equipment. Keep unprotected persons away.
· **Measures for environmental protection:** Do not allow to enter sewers/ surface or ground water.
· **Measures for cleaning/collecting:**
Absorb with liquid-binding material (sand, diatomite, acid binders, universal binders, sawdust).
Dispose contaminated material as waste according to item 13.
Ensure adequate ventilation.

## 7 Handling and storage

· **Handling:**
· **Information for safe handling:**
Ensure good ventilation/exhaustion at the workplace.
Open and handle receptacle with care.
· **Information about fire - and explosion protection:** Protect from heat.

· **Storage:**
· **Requirements to be met by storerooms and receptacles:** Prevent any seepage into the ground.
· **Information about storage in one common storage facility:** Store away from foodstuffs.
· **Further information about storage conditions:**
Store in cool, dry conditions in well sealed receptacles.
· **Storage class:**
· **Class according to regulation on flammable liquids:** Void

## 8 Exposure controls/personal protection

· **Additional information about design of technical facilities:** No further data; see item 7.

· **Ingredients with limit values that require monitoring at the workplace:**
The product does not contain any relevant quantities of materials with critical values that have to be monitored at the workplace.
· **Additional information:** The lists valid during the making were used as basis.

(Contd. on page 3)

**Trade name: EPOLAM 2050 DURCISSEUR**

(Contd. of page 2)

· **Personal protective equipment:**
· **General protective and hygienic measures:**
The usual precautionary measures are to be adhered to when handling chemicals.
Immediately remove all soiled and contaminated clothing
Wash hands before breaks and at the end of work.
Avoid contact with the eyes and skin.
· **Respiratory protection:** Not necessary if room is well-ventilated.
· **Protection of hands:**

Protective gloves

· **Material of gloves** PVC gloves
· **Eye protection:**

Safety glasses

Tightly sealed goggles
· **Body protection:** Protective work clothing

## 9 Physical and chemical properties

· **General Information**

| | |
|---|---|
| **Form:** | Fluid |
| **Colour:** | Amber coloured |
| **Odour:** | Amine-like |

· **Change in condition**

| | |
|---|---|
| **Melting point/Melting range:** | NA°C |
| **Boiling point/Boiling range:** | >200°C (DIN 53171) |
| · **Flash point:** | > 100°C (ISO 2719) |
| · **Ignition temperature:** | >300°C (DIN 51 794) |
| · **Decomposition temperature:** | >260°C (DIN 53171) |
| · **Self-igniting:** | Product is not selfigniting. |
| · **Danger of explosion:** | Product does not present an explosion hazard. |
| · **Density at 20°C:** | 0.96 g/cm³ |
| · **Solubility in / Miscibility with water:** | Not miscible or difficult to mix. |
| · **organic solvents:** | Soluble in many organic solvents. |
| · **pH-value at 20°C:** | >10 |

## 10 Stability and reactivity

· **Thermal decomposition / conditions to be avoided:**
No decomposition if used according to specifications.
· **Dangerous reactions** Strong exothermic reaction with acids.
· **Dangerous decomposition products:**
Corrosive gases/vapours

(Contd. on page 4)

**Trade name: EPOLAM 2050 DURCISSEUR**

(Contd. of page 3)

Ammonia

## 11 Toxicological information

· **Acute toxicity:**

| · **LD/LC50 values relevant for classification:** | | |
|---|---|---|
| **2855-13-2 3-aminomethyl-3,5,5-trimethylcyclohexylamine** | | |
| Oral | LD50 | 1030 mg/kg (rat) |
| Dermal | LD50 | 1840 mg/kg (rabbit) |
| **68479-98-1 diethylmethylbenzenediamine** | | |
| Oral | LD50 | 738 mg/kg (rat) |
| Dermal | LD50 | >2000 mg/kg (rat) |

· **Primary irritant effect:**
· **on the skin:** Caustic effect on skin and mucous membranes.
· **on the eye:** Strong caustic effect.
· **Sensitization:** Sensitization possible through skin contact.
· **Additional toxicological information:**
Swallowing will lead to a strong caustic effect on mouth and throat and to the danger of perforation of esophagus and stomach.

## 12 Ecological information

· **Information about elimination (persistence and degradability):**
· **Other information:** The product is difficultly biodegradable.
· **Ecotoxical effects:**

| · **Acquatic toxicity:** | |
|---|---|
| **2855-13-2 3-aminomethyl-3,5,5-trimethylcyclohexylamine** | |
| EC50 (24h) | 44 mg / l (daphnies) |
| EC50 (72h) | 37 mg/l (alga) |
| LC 50 (96h) | 110 mg / l (fish) |

## 13 Disposal considerations

· **Product:**
· **Recommendation**
Dispose of the product by burning in a suitable incinerator or bury in an approved landfield following all applicable local and/or national regulations.

| · **European waste catalogue** | |
|---|---|
| 20 01 27 | paint, inks, adhesives and resins containing dangerous substances |

· **Uncleaned packaging:**
· **Recommendation:**
Empty containers may not be disposed of unless any remaining material adhering to the internal walls has been removed.
Disposal must be made according to official regulations.
· **Recommended cleansing agents:** Water, if necessary together with cleansing agents.



(Contd. on page 5)

**Trade name: EPOLAM 2050 DURCISSEUR**

(Contd. of page 4)

## 14 Transport information

· **Land transport ADR/RID (cross-border)**

· **ADR/RID class:** 8 Corrosive substances.
· **Danger code (Kemler):** 80
· **UN-Number:** 1760
· **Packaging group:** III
· **Hazard label** 8
· **Description of goods:** 1760 CORROSIVE LIQUID, N.O.S. (Polyoxyalkyleneamine)

· **Maritime transport IMDG:**

· **IMDG Class:** 8
· **UN Number:** 1760
· **Label** 8
· **Packaging group:** III
· **EMS Number:** 8-15
· **Marine pollutant:** No
· **Proper shipping name:** CORROSIVE LIQUID, N.O.S. (Polyoxyalkyleneamine)

· **Air transport ICAO-TI and IATA-DGR:**

· **ICAO/IATA Class:** 8
· **UN/ID Number:** 1760
· **Label** 8
· **Packaging group:** III
· **Proper shipping name:** CORROSIVE LIQUID, N.O.S. (Polyoxyalkyleneamine)

## 15 Regulatory information

· **Labelling according to EU guidelines:**
The product has been marked in accordance with EU Directives / respective national laws.

· **Code letter and hazard designation of product:**
C Corrosive
N Dangerous for the environment

· **Hazard-determining components of labelling:**
3-aminomethyl-3,5,5-trimethylcyclohexylamine
diethylmethylbenzenediamine
Polyoxyalkyleneamine

· **Risk phrases:**
21/22 Harmful in contact with skin and if swallowed.
34 Causes burns.
43 May cause sensitisation by skin contact.
48/22 Harmful: danger of serious damage to health by prolonged exposure if swallowed.

(Contd. on page 6)

(Contd. of page 5)

51/53 Toxic to aquatic organisms, may cause long-term adverse effects in the aquatic environment.

· **Safety phrases:**

3 Keep in a cool place.
20 When using do not eat or drink.
26 In case of contact with eyes, rinse immediately with plenty of water and seek medical advice.
36/37/39 Wear suitable protective clothing, gloves and eye/face protection.
45 In case of accident or if you feel unwell, seek medical advice immediately (show the label where possible).
57 Use appropriate container to avoid environmental contamination.

· **National regulations:**

· **Classification according to VbF:** Void

· **Waterhazard class:** Water danger class 3 (Self-assessment): extremely hazardous for water.

## 16 Other information

This information is based on our present knowledge. However, this shall not constitute a guarantee for any specific product features and shall not establish a legally valid contractual relationship.

· **Relevant R-phrases**

21/22 Harmful in contact with skin and if swallowed.
34 Causes burns.
36 Irritating to eyes.
43 May cause sensitisation by skin contact.
48/22 Harmful: danger of serious damage to health by prolonged exposure if swallowed.
50/53 Very toxic to aquatic organisms, may cause long-term adverse effects in the aquatic environment.
52/53 Harmful to aquatic organisms, may cause long-term adverse effects in the aquatic environment.

· **Department issuing MSDS:** Product Safety and Toxicology